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SUPERFUND TREATABILITY CLEARINGHOUSE

Document Reference:

EBASCO Services Inc. "Litigation Technical Support and Services, Rocky Mountain Arsenal (Basis F Wastes)." Six-part technical report with a total of approximately 600 pp. prepared for U.S. Army Program Manager's Office for Rocky Mountain Arsenal Cleanup during April and September 1986 and March, April, and May 1987.

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Superfund Treatability Clearinghouse - FDBP

SUPERFUND TREATABILITY CLEARINGHOUSE ABSTRACT

Treatment Process:	Thermal Treatment - Incineration
Media:	Soil/Generic
Document Reference:	EBASCO Services Inc. "Litigation Technical Support and Services, Rocky Mountain Arsenal (Basis F Wastes)." Six-part technical report with a total of approximately 600 pp. prepared for U.S. Army Program Manager's Office for Rocky Mountain Arsenal Cleanup during April and September 1986 and March, April, and May 1987.
Document Type:	Contractor/Vendor Treatability Study
Contact:	Bruce Huenfeld U.S. DOD/USATHAMA Aberdeen Proving Ground, MD 21010-5401 301-617-3446
Site Name:	Rocky Mountain Arsenal, CO (NPL - Federal facility)
Location of Test:	Rocky Mountain, CO

BACKGROUND: This report consists of 5 documents which cover incineration tests at the Rocky Mountain Arsenal (RMA), Denver, CO, ranging from a laboratory test plan and bench-scale test to full-scale testing. This abstract reports only on the results of bench-scale incineration tests of contaminants from Basin F of the RMA. Objectives of the study were to: 1) Gather information on properties of the wastes, 2) provide a bench-scale apparatus to determine incinerability characteristics of the wastes, 3) demonstrate 99.99% destruction removal efficiency (DRE), and 4) determine gas residence time, temperature and excess O_2 necessary for 99.99% DRE.

OPERATIONAL INFORMATION: The types of waste discharged into the Basin F lagoon included sodium salts of chloride, fluoride, hydroxide, methyl phosphate, acetate, sulfate and pesticides.

Bench-scale tests were conducted on pure compounds and field samples. The technical approach involved using equipment to simulate three of the major incineration mechanisms--pyrolysis, primary incinerator postflame, and afterburner postflame.

The laboratory bench-scale unit was designed to evaluate thermal destruction efficiency up to 1200° F and residence times from 2 to 5 seconds. The unit utilized a batch load system with two furnaces and a blended carrier gas. The first furnace volatilized the constituents while the carrier gas moved the constituents to the secondary furnace which added O_2 and simulated an afterburner in a full-scale unit.

Residence times in the afterburner were 1 second or 5 seconds. Residence time in the primary burner was one hour. Temperature parameters for the primary and secondary chambers were based on the current limitations of operational practices for waste incineration. Primary burner operating temperatures were 650°, 800° and 900o C. Secondary afterburner operating

NOTE: Quality assurance of data may not be appropriate for all uses.

temperatures were 650°, 900° and 1200° C. O_2 concentrations were 5% to 7%. Sixteen successful runs were performed.

The combustion products in the gases were collected by a sampling train for subsequent analysis. A detailed sampling plan is contained in this study. An outline of QA/QC measures that will be taken are reported in the "Draft Laboratory Test Plan for Incineration of Basin F Wastes at Rocky Mountain Arsenal, April 1986." Samples for analysis were collected from soils, sludge and liquid. GC/MS was employed to analyze for ten semivolatile compounds in the feed stock. GS/MS selective ion monitoring was used for contaminant residue and off gas analysis.

PERFORMANCE: In all but a few instances, a 99.99% DRE was demonstrated for the ten principal hazardous organic constituents. Residues were tested for EP Toxicity to determine the leachability of heavy metals contained in the Basin F wastes. No heavy metals exceeded the EP Toxicity limit. In summary, Basin F wastes are incinerable and DRE levels were 99.99% under almost all the conditions investigated.

CONTAMINANTS:

Analytical data is provided in the treatability study report. The breakdown of the contaminants by treatability group is:

Treatability Group	CAS Number	Contaminants
W01-Halogenated Non-Polar Aromatic Compounds	108-90-7	Chlorobenzene
W03-Halogenated Phenols Cresols and Thiols	CPMS	P-Chlorophenylmethyl Sulfide
	CPMSO2	P-Chlorophenylmethyl Sulfone
	CPMSO	P-Chlorophenylmethyl Sulfoxide
	470-90-6	Supona
W04-Halogenated Aliphatic Solvents	96-12-8	1,2-Dibromo-3-chloropropane
W05-Halogenated Cyclic Aliphatics/Ethers/ Esters/Ketones	309-00-2	Aldrin
	72-20-8	Endrin
	465-73-6	Isodrin
	60-57-1	Dieldrin
W07-Hetercyclics and Simple Aromatics	108-88-3	Toluene
	1330-20-7	Xylenes
	ABC	Alkyl Benzene
W09-Other Polar Organic Compounds	109-92-2	Ethoxyethylene
	110-71-4	Dimethoxyethane
	T119-36-8	Benzoic Acid
W13-Other Organics	142-82-5	Heptane
	77-73-6	Dicyclopentadiene

Note: This is a partial listing of data. Refer to the document for more information.

NOTE: Quality assurance of data may not be appropriate for all uses.

TABLE 1

DESTRUCTION AND REMOVAL EFFICIENCY OF TEN PRINCIPAL HAZARDOUS ORGANIC CONSTITUENTS IN OVERBURDEN SAMPLE

Temp Degrees C in Secondary Burner	650	650	650	900	900	900	900	900	900	900	1200	1200	1200	1200
Temp Degrees C in Primary Burner	650	650	650	650	800	800	900	900	900	900	650	900	900	900
Gas Residence Time in Second Burner (in seconds)	2	2	5	2	2	5	2	2	5	5	5	2	2	5
Oxygen Level in off-gas (%)	5.4	7	7	5.4	7	7	5.4	7	5.4	7	5.4	5.4	7	7
Run Number	14 17	11	6	18	20	18	12	3	9	7	8	10 13	2	5
ALDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.94	100.00	100.00	100.00	100.00 100.00	100.00	100.0
CPMS	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00 100.00	100.00	100.0
CPMSO	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.41	100.00	100.00	100.00	100.00 100.00	100.00	100.0
CPMSO2	100.00 100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.0
DBCP	99.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.0
DIELDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.97	100.00	100.00	100.00	100.00 100.00	100.00	100.0
ENDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.0
ISODRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00 100.00	100.00	100.0
SUPONA	99.74 100.00	99.38	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.0

Document Number: FDBP

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980-TS1-RT-FDBP

DRAFT

BENCH-SCALE LABORATORY INCINERATION OF BASIN F WASTES ROCKY MOUNTAIN ARSENAL

MAY 1987

TASK NO. 17

CONTRACT NO. DAAK11-84-D-0017

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BENCH-SCALE LABORATORY INCINERATION
OF
BASIN F WASTES

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TEXT ABBREVIATIONS

A	:	Frequency Factor
°C	:	Degree Celsius
DRE	:	Destruction and Removal Efficiency
E_a	:	Activation Energy
EP	:	Extraction Procedure
FID	:	Flame Ionization Detector
GC-MS	:	Gas Chromatography - Mass Spectrometry
MM5	:	Modified Method 5
PCT	:	Physical, Chemical and Thermodynamic
PIC	:	Products of Incomplete Combustion
PMO	:	Program Manager's Office for Rocky Mountain Arsenal Contamination Cleanup
POHCs	:	Principal Organic Hazardous Constituents
RCRA	:	Resource Conservation and Recovery Act
SIM	:	Selective Ion Monitoring
SWLP	:	Solid Waste Leaching Procedures
T	:	Temperature
cm	:	centimeter
ft	:	foot or feet
gm	:	gram
kcal	:	Kilocalorie
kg	:	kilogram
l	:	liter
lb	:	pound
mg	:	milligram
mm	:	millimeter
ppb	:	parts per billion
ppm	:	parts per million
s	:	second
$\bar{t}_r$	:	gas residence time
ug	:	microgram

CHEMICAL ABBREVIATIONS

Ag	:	Silver
As	:	Arsenic
Ba	:	Barium
BCHD	:	Bicycloheptadiene
Ca	:	Calcium
Cd	:	Cadmium
$CHCl_3$	:	Chloroform
CO_2	:	Carbon Dioxide
COD	:	Chemical Oxygen Demand
CPMS	:	P-Chlorophenylmethyl Sulfide
CPMSO	:	P-Chlorophenylmethyl Sulfoxide
$CPMSO_2$	:	P-Chlorophenylmethyl Sulfone
Cr	:	Chromium
Cu	:	Copper
DBCP	:	Nemagon (Dibromo Chloropropane)
DCPD	:	Dicyclopentadiene
DDE	:	1,1-dichloro-2, 2-bis-(p-chlorophenyl) ethylene
DDT	:	Dichloro diphenyl trichloroethane
DIMP	:	Diisopropylmethyl Phosphonate
DMDS	:	Dimethyl Disulfide
DMMP	:	Dimethymethyl Phosphate
H	:	Hydrogen
H_2O	:	Water
HBr	:	Hydrogen Bromide
HCB	:	Hexachloro-1, 3-butadiene
HCl	:	Hydrogen Chloride
HCCPD	:	Hexachlorocyclopentadiene
Hg	:	Mercury
K	:	Potassium
Mg	:	Magnesium
MIBK	:	Methyl Isobutyl Ketone
N_2	:	Nitrogen
Na	:	Sodium

CHEMICAL ABBREVIATIONS

(Continued)

NaOH	:	Sodium Hydroxide
O_2	:	Oxygen
OH	:	Hydroxyl
Pb	:	Lead
PERC	:	Tetrachloroethene
PNA	:	Polynuclear Aromatic
Se:	:	Selenium
TOC	:	Total Organic Carbon
Zn	:	Zinc

1.0 INTRODUCTION

1.1 PURPOSE

The Program Manager's Office for Rocky Mountain Arsenal Contamination Cleanup (PMO) is currently in the process of gathering information on the technical and economic aspects of incineration/thermal treatment of Basin F wastes. This information gathering process is one aspect of developing a broad remedial action alternative for Basin F. The PMO has taken this action in accordance with the National Contingency Plan, 50 Federal Register 47912 (1985). Accordingly, the PMO has contracted Ebasco to conduct this work effort under Task Order 17.

Task Order 17 comprises several distinctly separate work elements (Ebasco, 1986a). One of these work elements consists of laboratory investigations for determining the incinerability characteristics of Basin F wastes. Under this work element, Ebasco has designed and executed a laboratory test program (Ebasco, 1986b). This report describes the rationale, performance, and results of that undertaking.

The objectives of the laboratory test program were to:

- Gather information on the physical, chemical, and thermodynamic properties of Basin F wastes (both liquid and contaminated soils) to ensure reasonable success in designing an incineration program;

- Provide a bench-scale apparatus that can be used to determine the incinerability characteristics of Basin F wastes;

- Demonstrate that 99.99 percent destruction and removal efficiency (DRE) is achievable for the hazardous organic constituents associated with Basin F wastes;

- Determine what residence times, temperatures, and levels of excess oxygen (O_2) will achieve 99.99 percent DRE within the most cost-effective incinerator technology framework; and

- Provide guidance for selecting the final incineration technology and optimizing the transition from a bench-scale system to a pilot plant, or from a bench-scale system to a full-scale operation.

1.2 STUDY CONSTRAINTS

The laboratory test program was designed within the available resources. The basic objective of the program was to determine and demonstrate that contaminants associated with Basin F soils and liquids could be decomposed through thermal treatment. To that extent, the program was delineated by the following factors:

- The bench-scale thermal destruction device was constructed to determine the thermal decomposition characteristics of Basin F contaminants in a nonflame mode environment;

- Testing of Basin F wastes was limited to contaminated solids (overburden) overlying the basin's asphalt liner and liquid from the main impoundment;

- Maximum temperatures at which the bench-scale incineration system was operated were 900°C at the primary burner and 1200°C at the secondary burner;

- Secondary burner residence times were 2 and 5 seconds; and

- Maximum primary burner sample sizes were limited to 500 grams.

1.3 STUDY APPROACH

The technical approach developed for this program recognizes the inherent limitations of investigations within a laboratory environment as well as the lack of precise data concerning the feedstocks to be incinerated.

The technical approach involves using equipment to simulate three of the major incineration mechanisms: (1) pyrolysis; (2) primary incinerator post-flame; and (3) afterburner postflame.

The technical approach was designed to focus on and evaluate the impacts of incinerating Basin F contaminated soil. Initially, this approach did not designate one or more principal organic hazardous constituents (POHCs) to be incinerated, but rather evaluated the impact of incineration on all compounds identified in the soil samples.

The technical approach began with limited characterization of selected compounds in terms of physical, chemical, and thermodynamic properties. Following this characterization, the contaminated soil compounds were then tested for determining the impacts from incineration at two residence times (in the afterburner), two temperatures, and two levels of excess O_2. Multiple runs were used to ensure that the DRE associated with any compound would not be masked, regardless of the concentration of the incoming material to be incinerated.

2.0 CHARACTERISTICS OF BASIN F WASTES

2.1 INTRODUCTION

Waste characterization is a major factor in assessing the feasibility of destroying hazardous waste material by incineration. This characterization affects the design of the incinerator and its emission control system, and aids in determining incinerator operating conditions for complete destruction of a specific organic compound.

This chapter discusses physical, chemical, and thermodynamic (PCT) properties of Basin F wastes that are important in evaluating the incineration technology as well as designing a full-scale incineration system based on the selected incineration technology. The discussion of PCT properties of Basin F wastes are based on past studies and limited sampling and analyses performed under this program (Task 17).

2.2 BACKGROUND

2.2.1 Basin F History

Basin F is located in the northwest part of the Rocky Mountain Arsenal in Section 26. This asphalt-lined basin had been used for total retention of chemical wastes generated from Army and Shell operations. The basin was used from 1956 to 1982. The types of chemical wastes discharged into the basin consisted mainly of aqueous solutions of various sodium salts including chloride, fluoride, hydroxide, methyl phosphonate, acetate, sulfate, and pesticides. The potential for industrial waste discharge into Basin F was eliminated in 1982 when the chemical sewer line feeding the basin was excavated. The remaining Basin F liquid has been evaporating since that time. A comprehensive study conducted in 1982 revealed that the overburden and soil underneath the liner of the basin have been contaminated with various chemicals that had accumulated in Basin F during its operational period.

2.2.2 Basin F Waste Characteristics

In addition to the actual liquid wastes contained within Basin F, three other categories of materials are present which may be considered waste materials. These are the basin liner itself, the overburden above the liner (including precipitates), and any contaminated soil adjoining the basin or beneath the liner. Overburden, liner, and contaminated soils can be considered together for treatment and disposal.

Numerous analyses have been conducted on Basin F liquid through the years. A comprehensive review of the previous analytical results was conducted in 1977 (Buhts et al., 1977). The results of this effort are summarized in Table 2.2-1. Contaminant concentrations in the liquid have likely increased since 1977 due to the evaporation of water within the basin.

A comprehensive study of Basin F was conducted in 1982 to determine the distribution of contaminants in the overburden and in the soil underlying the liner, and to assess the condition of the liner (Myers & Thompson, 1982). This study involved 16 shallow borings in the exposed portion of the basin as indicated in Figure 2.2-1.

The sample cores and samples of the overburden were subjected to a series of analytic extraction procedures. Among those initially considered were extraction procedure (EP) toxicity, solid waste leaching procedures (SWLP), and total extraction (bulk analysis). The EP toxicity procedure yields a determination of whether the waste would be considered hazardous under the Resource Conservation and Recovery Act (RCRA). The SWLP is similar to the EP toxicity test with the exception that a neutral pH water is used as an extract to more accurately simulate leachate migration potential (Myers & Thompson, 1982). Bulk analyses utilize a solvent rinse to determine the gross amount of contaminant held within the waste matrix that could be potentially released.

The extracts from the SWLP tests conducted on subsamples of the cores were analyzed for a select group of contaminants that had been identified previously in the Basin F liquid. The concentrations of many of the

TABLE 2.2-1

CHEMICAL CHARACTERIZATION OF BASIN F LIQUID

Compound or Parameter	Units	Concentration Range[1/]
pH	-	6.9 - 7.2
Aldrin	ppb	50.0 - 400
Isodrin	ppb	2.0 - 15
Dieldrin	ppb	5.0 - 110
Endrin	ppb	5.0 - 40
Dithiane	ppb	30.0 - 100
DIMP	ppm	10.0 - 20
DMMP	ppm	500.0 - 2,000
Sulfoxide	ppm	4.0 - 10
Sulfone	ppm	25.0 - 60
Chloride	ppm	48,000.0 - 56,000
Sulfate	ppm	21,000.0 - 25,000
Copper	ppm	700.0 - 750
Iron	ppm	5.0 - 6
Nitrogen	ppm	120.0 - 145
Phosphorus (total)	ppm	2,050.0 - 2,150
Hardness	ppm	2,100.0 - 2,800
Fluoride	ppm	110.0 - 117
Arsenic	ppm	1.0 - 1.3
Magnesium	ppm	35.0 - 40
Mercury	ppb	26.0 - 29
Cyanide	ppm	1.45 - 1.55
COD	ppm	24,500.0 - 26,000
TOC	ppm	20,500.0 - 22,500

1/ Based on analysis of various samples from different locations and depths in the basin (Buhts et al., 1977).

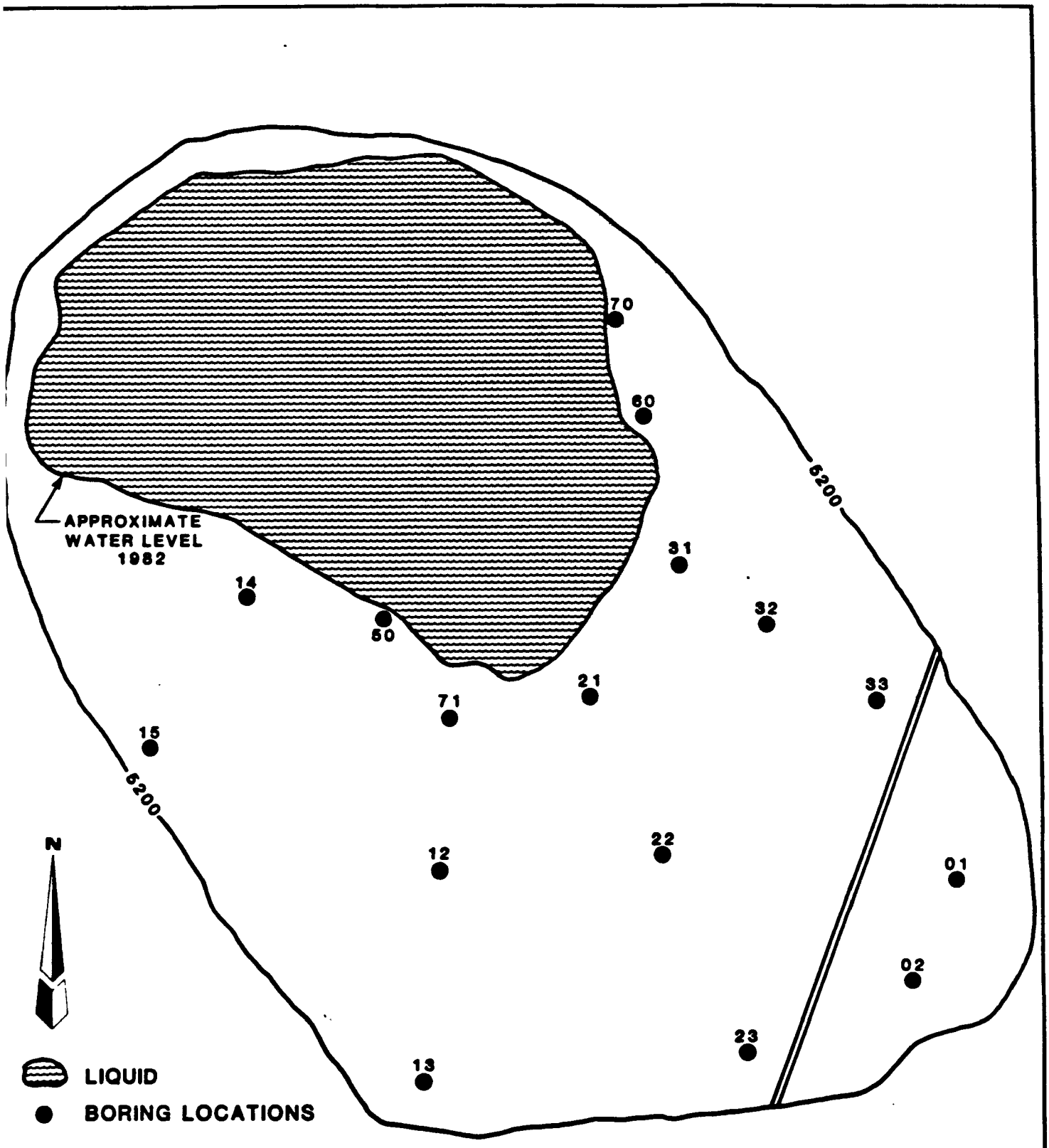


FIGURE 2.2-1
LOCATION OF BORING SITES WITHIN BASIN F

SOURCE: MYERS AND THOMPSON, 1982

contaminants in the SWLP extracts were very low or below detectable limits (Myers & Thompson, 1982). A map summarizing the SWLP cores is presented in Figure 2.2-2.

The contaminants found above their respective action level concentrations included Aldrin, Dieldrin, Endrin, Isodrin, organo-sulfur compounds, dibromochloropropane (DBCP), arsenic, and fluoride. Borings 01 and 02 were found to have the greatest number of contaminants in the extracts for all depths intervals. Also, the concentrations of the contaminants in the extracts from these two borings were generally higher than those associated with other borings.

The SWLP tests, conducted on the overburden samples collected from five boring sites, resulted in much higher concentrations of contaminants in the extracts than in those associated with the soils underlying the liner. In addition to the contaminants identified in the SWLP extracts from the cores, concentrations of diisopropylmethyl phosphate (DIMP) and dicyclopentadiene (DCPD) were found in some of the extracts from the overburden.

Only the extracts from the cores collected at Boring 02 from the 0 to 1 foot (ft) and 1 to 2 ft intervals exhibited concentrations exceeding 100 times their respective water quality levels (see Figure 2.2-2). For the 0 to 1 ft interval, the concentrations of Aldrin, Dieldrin, Endrin, and Isodrin in the extract exceeded the criteria. In the 1 to 2 ft interval, only the concentration of Dieldrin in the extract exceeded the criteria.

As discussed previously, Boring 02 was the only boring location in the study where the liner was found to be in poor condition. Contamination in the overburden or liquid (when this area was innundated) probably was able to migrate in high concentrations into the soil due to the deteriorated condition of the liner. In other areas of the basin evaluated in this study, the liner appeared to have maintained sufficient integrity to prevent the migration of large amounts of contaminants into the underlying soils.

Recent investigation (ESE, 1986) at Basin F revealed that subsurface soil contamination has occurred only in locations where integrity of the asphalt

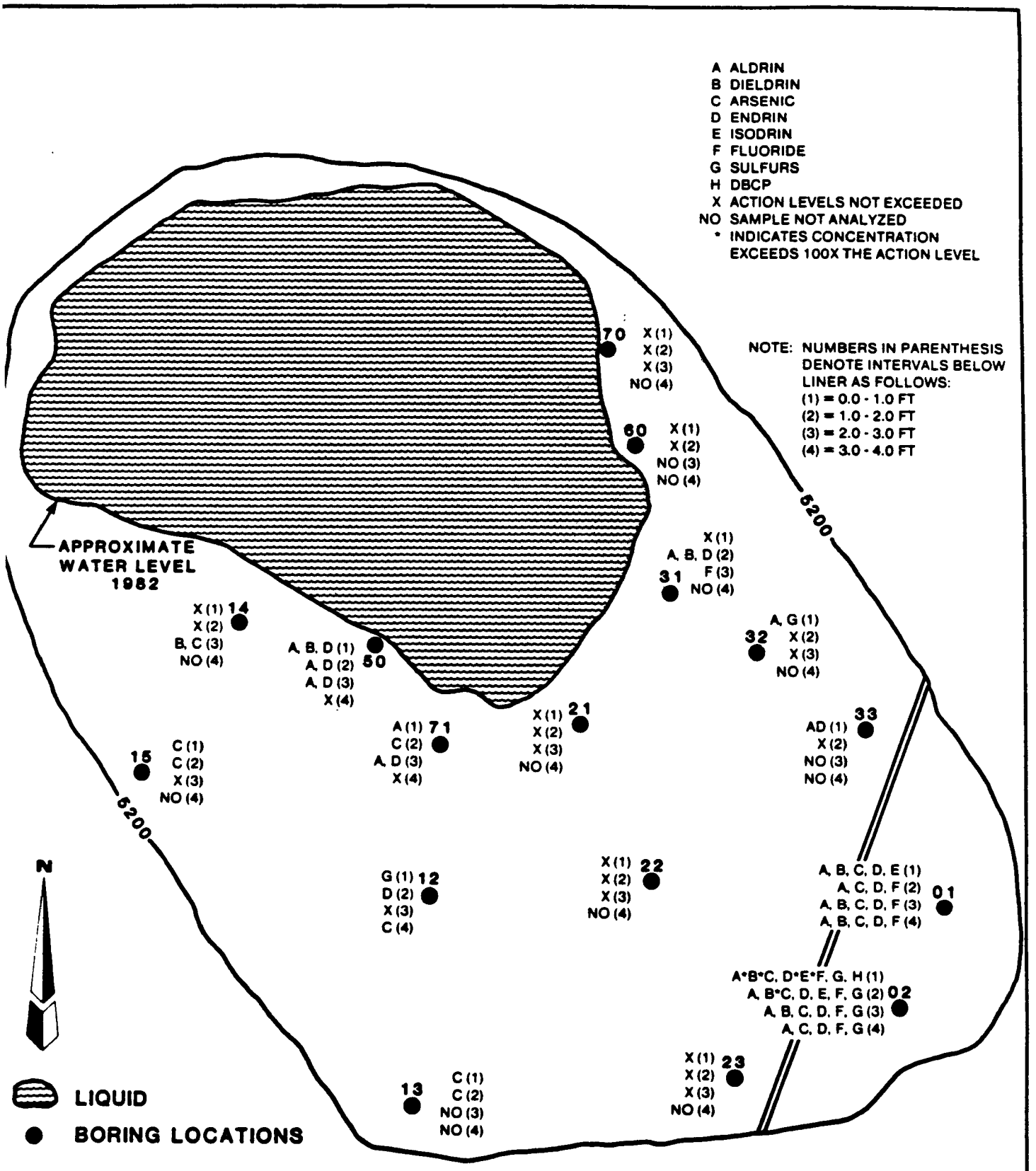


FIGURE 2.2-2
CONTAMINANTS IDENTIFIED IN THE SWLP EXTRACTS
OF THE SOILS IN BASIN F

OURCE: MYERS AND THOMPSON, 1982

liner is questionable. Analytical data collected from this investigation is presented in Table 2.2-2. The study concluded that the highest levels of contaminants (a variety of volatile organics, organochlorine pesticides, and elevated levels of metals) were found in the boreholes located along the eastern boundary of the basin and/or in areas where liner integrity was poor (ESE, 1986). Deepest contamination (35 ft) was found along the east boundary and in the southernmost area of Basin F.

2.3 SAMPLING

The purpose of the sampling program was to collect Basin F waste materials in sufficient quantities for bench-scale thermal destruction testing. The sampling program was not designed to collect representative samples, i.e., samples that represent the average spectrum of contaminants associated with Basin F.

Grab samples from the north end of the basin were collected using a bucket-type sampler. Approximately 15 gallons of liquid was collected during the liquid sampling program.

Soil sampling was limited to collecting overburden from the most contaminated area of the basin. It was determined that the source contamination problems should be tested explicitly. For that reason, overburden from the Borehole 01 was collected by using a hand auger. Borehole 01 was selected for sampling because past study has indicated highest concentrations of contaminants in this borehole than any other boreholes studied. Approximately 30 kilograms of overburden sample were collected during the soil sampling program.

2.4 LABORATORY ANALYSIS

2.4.1 Objectives

The Basin F wastes characterization program was designed to gather information on physical, chemical, and thermodynamic properties that are essential in understanding the performance of the bench-scale thermal

CONCENTRATIONS OF CONTAMINANTS IN SOIL SAMPLES UNDERLYING BASIN F LINER

Constituents	Number of Detections*	Concentrations (ug/gm) Range	Mean	Median	Standard Deviation	ESE Detection Limit (ug/gm)	MRI Detection Limit (ug/gm)
Volatiles (N=40)†							
Chlorobenzene	2	0.8-5	3	3	3	0.3	0.3
$CHCl_3$	3	0.3-70	30	4	40	0.3	0.7
1,2-Dichloroethane	1	1	—	—	—	0.3	0.4
BCHD	5	2-30	9	5	10	0.3	0.8
BCHD	1	25	—	—	—	—	—
Ethylbenzene	2	1-8	5	5	5	0.3	0.4
Tetrachloroethene	7	1-40	10	10	10	0.3	0.5
Tetrachloroethene	1	25	—	—	—	—	—
Toluene	7	1-1000	400	300	400	0.3	0.3
Toluene	—	25	—	—	—	—	—
1,1,1-Trichloroethane	1	0.4	—	—	—	0.3	0.5
m,xylene	2	0.4-5	2	2	3	0.3	—
MIBK	2	0.4-1	0.7	0.7	0.4	0.5	0.4
DMDS	3	2-60	30	10	30	0.3	4.0
Benzene	3	1-3	2	2	1	0.3	1.0
o,p-xylene	1	10	—	—	—	0.5	0.5
Semi-Volatiles (N=40)							
Aldrin	9	0.7-4000	1000	1000	1000	0.9	0.5
Dieldrin	7	100-2000	500	400	500	0.3	0.6
Endrin	7	90-900	500	400	300	0.7	4.0
DIMP	2	0.5-0.8	0.6	0.7	0.2	0.5	3.0
Isodrin	7	100-3000	1000	1000	1000	0.3	0.6
DCPD	7	30-4000	1000	600	1000	0.3	6.0
DBCP	7	0.044-8.1	2.4	0.86	3.0	0.005	0.005
PCPMS	3	6-700	400	400	400	0.3	0.3
PCPMSO	5	4-70	20	5	30	0.4	1.0
DMMP	6	3-70	20	7	30	2	3.0
$PCPMSO_2$	14	0.5-300	30	5	70	0.3	0.4
Metals (N=40)							
Cadmium	1	2.0	—	—	—	0.9	0.5
Chromium	36	11-34	19	18	5.6	7.2	7.4
Copper	40	5.0-2300	85	16	370	4.8	4.9
Lead	4	18-35	24	21	7.7	17	16
Zinc	35	33-320	68	57	49	16	28
Arsenic	20	4.8-18	9.6	9.2	3.6	4.7	5.2
Mercury	1	0.08-0.08	0.08	0.08	0.08	0.05	0.07

* Number of samples in which constituent was detected.
† N = Number of samples analyzed.

Source: ESE (1986).

destruction unit and also in designing a full-scale incineration system. It should be recognized that the sampling and analytical programs were limited in scope, and as such, the results derived from these programs may not be representative of the entire spectrum of wastes associated with Basin F.

2.4.2 Analytical Parameters

The physical, chemical, and thermodynamic parameters that were analyzed for Basin F liquid and overburden samples are identified in Table 2.4-1.

2.4.3 Basin F Liquid

The Basin F liquid sample was described to have a motor oil-like appearance and a fairly homogeneous composition. The pH of the sample was 6.02. The analytical results of the Basin F liquid sample are shown in Tables 2.4-2 and 2.4-3. Only parameters found in concentrations higher than their detection limits are reported in Tables 2.4-2 and 2.4-3. Table 2.4-2 presents the results of proximate, heating value, and ash fusion temperature analyses of Basin F liquid and overburden samples.

Basin F liquid is highly corrosive (corrosivity 50 millimeters per year). It is neither ignitable nor reactive. The heating value of the liquid waste was found to be 4 Btu/lb. Table 2.4-3 presents analytical results of organic, inorganic, and metal constituents that were detected in Basin F liquid.

The concentration of sodium (Na) was found to be 2300 parts per million (ppm) while calcium (Ca), magnesium (Mg) and potassium (K) ranged from 5 to 30 ppm. Among the inorganic constituents, chloride concentration was found to be the highest (120,000 ppm) which is almost 2.5 times more than what was found in 1977 (See Table 2.2-1).

Among the trace metals, copper concentration was found to be high (210 ppm) while cadmium (Cd), lead (Pb), and mercury (Hg) were in the parts per billion (ppb) range (74 to 140 ppb).

TABLE 2.4-1

SUMMARY OF LABORATORY ANALYSES

Overburden (One Sample)

- Volatiles by GC/MS
- Semi-Volatiles by GC/MS
- ICP Metals
- Arsenic
- Mercury
- 3 RCRA Tests (ignitability, reactivity, corrosivity)
- Proximate and Ultimate Analysis
- Elemental Composition
- Heating Value

Liquid (One Sample)

- Volatile Aromatics
- Volatile Halocarbons
- Organo Chlorine Pesticides
- DCPD/BCHPD/MIBK
- Organo Sulfur Compounds
- DIMP/DMMP
- ON/OP Pesticides
- Arsenic
- Mercury
- ICP Metals
- 3 RCRA Tests
- Elemental Composition
- Heating Value

TABLE 2.4-2

PHYSICAL, CHEMICAL, AND THERMODYNAMIC TEST RESULTS
OF BASIN F LIQUID AND OVERBURDEN [1/]

Parameter	Liquid	Overburden
Proximate Analysis (percent weight, wet)		
Moisture		23.93
Volatiles		14.47
Fixed Carbon		2.33
Ash		59.20
		100.01
Higher Heating Value (Btu/lb)		
Wet Basis	4	37
Ash Fusion Temperature (°F)[2/]		
Initial Deformation		2242 - 2253
Softening		2329 - 2377
Hemispheric Lump		2512 - 2622
Fluid		2784 - 3000
Reactivity (mg/gm)		
Cyanide	<0.02	3.6
Sulfide	<0.02	<0.02
Corrosivity		
Millimeters/year	50	1.1
Other		
Bulk Density (lbs/ft^3)		100
Specific Heat Dry (Btu/lb-°F)		0.25 - 0.30
Thermal Conductivity (Btu/hr-ft-°F)		0.32

1/ Tests were conducted by UBTL in Salt Lake City under Task 17.

2/ On one of the ash fusion analyses, a eutectic effect was indicated.

TABLE 2.4-3

ANALYTICAL RESULTS OF BASIN F LIQUID SAMPLE

Organics		Inorganics	
Aldrin (ppb)	2,300	Calcium (ppm)	6.8
Dieldrin (ppb)	459	Magnesium (ppm)	5.6
Endrin (ppb)	596	Potassium (ppm)	30
Isodrin (ppb)	1,980	Sodium (ppm)	2,300
ppDDE (ppb)	109	Chloride (ppm)	120,000
ppDDT (ppb)	340	Fluoride (ppm)	21
DIMP (ppb)	400		
CPMSO (ppb)	1,000	METALS	
$CPMSO_2$ (ppb)	1,000	Arsenic (ppm)	3.0
Hexachlorocylopentadiene (ppb)	1,850	Cadmium (ppb)	8.4
Atrazine (ppb)	220	Chromium (ppb)	85
Malathion (ppb)	810	Copper (ppm)	210
Parathion (ppb)	110	Lead (ppb)	74
Supona (ppb)	340	Mercury (ppb)	140
Vapona (ppb)	890	Zinc (ppb)	950
Benzene (ppb)	7.7		

The Basin F liquid analysis for selected organic compounds indicated no volatile halogenated organics present above the limits of analytical detection. Of the volatile aromatic organics, benzene was found to be 7.7 ppb.

Among the organochlorine pesticides, Aldrin, Isodrin, and hexachlorocylopentadiene were found to be 2300 ppb, 1980 ppb, and 1850 ppb, respectively. The remaining chlorinated pesticides ranged from 109 to 596 ppb.

The organophosphate pesticides ranged from 110 to 890 micrograms per liter (µg/l) or ppb, with highest concentration being attributed to Vapona (890 ppb).

The concentration of DIMP, CPMSO, and $CMPSO_2$ were found to be 400 ppb, 1000 ppb, and 1000 ppb, respectively.

2.4.4 Basin F Overburden

The analytical results of the overburden sample are shown in Tables 2.4-2 and 2.4-4. The proximate analysis showed that the sample contained almost 24 percent moisture and 15 percent volatile matters. The ash content of the sample was found to be 59 percent. The heating value of the sample was measured to be 37 Btu/lb.

Analyses on reactivity, corrosivity, and ignitability indicated the overburden sample to be nonignitable, corrosive (corrosivity 1.1 millimeter per year), and reactive due to cyanide content (3.6 milligrams per gram).

Concentrations of sodium and chloride were found to be 4500 ppm and 1700 ppm, respectively. Among the trace metals, the concentration of copper was found to be the highest (5900 ppm), followed by zinc (430 ppm) and lead (270 ppm).

The organic analyses indicated an absence of any volatile species. The chlorinated pesticides present in the sample were Aldrin, Dieldrin, Endrin,

TABLE 2.4-4

ANALYTICAL RESULTS OF THE OVERBURDEN SAMPLE

Organics		Inorganics	
Aldrin (ppm)	2480	Sodium (ppm)	4500
Dieldrin (ppm)	1300	Chloride (ppm)	1700
Endrin (ppm)	165		
Isodrin (ppm)	100	METALS	
Supona (ppm)	6.7		
DCPD (ppm)	69.7	Arsenic (ppm)	3.9
DBCP (ppm)	13.7	Cadmium (ppm)	1.4
CPMS (ppm)	216	Chromium (ppm)	83
CPMSO (ppm)	34.7	Copper (ppm)	5900
$CPMSO_2$ (ppm)	180	Lead (ppm)	270
		Mercury (ppm)	3.5
		Zinc (ppm)	430

and Isodrin. Concentrations of Aldrin and Dieldrin were found to 2480 ppm and 1300 ppm, respectively.

The other organic compounds found in the sample were DCPD, DBCP, CPMS, CPMSO, and $CPMSO_2$ with concentrations ranging from 13 to 216 ppm.

3.0 BENCH-SCALE INCINERATION SYSTEM

3.1 RATIONALE FOR A BENCH-SCALE TEST SYSTEM

Thermal decomposition laboratory tests have been performed on both pure compounds and field samples to determine incineration parameters, including residence time, temperature, and excess oxygen required to decompose toxic chemicals. These laboratory tests have been performed primarily using milligram-to-gram size samples. These small sample sizes have been adequate to characterize incineration parameters for pure compounds and compounds in high concentrations. For chemicals that are present in low concentrations, these small sample sizes have not been adequate to demonstrate 99.99 percent destruction due to the analytical limits of detection of the off-gases. It is of interest to demonstrate 99.99 percent destruction for all toxic constituents in a feed sample regardless of whether or not that constituent is chosen as a POHC. Although there are substantial data on the thermal destruction of individual compounds, incineration tests on field samples are necessary to adequately simulate the interaction of various constituents at high temperatures and the production of products of incomplete combustion (PIC). The bench-scale test unit for Task 17 was designed to measure DRE up to 99.99 percent for constituents of concern at Basin F for soil, sludge, and liquid samples.

3.2 SYSTEM DESCRIPTION

The laboratory bench-scale unit was designed to evaluate thermal destruction efficiency at temperatures up to 1200°C and residence times from 2 to 5 seconds. The unit utilizes a batch-load system with two furnaces and a blended carrier gas to simulate combustion gases (Figure 3.2-1). The first furnace volatilizes the constituents while the carrier gas moves these constituents into the secondary furnace which simulates afterburners in a full-scale incineration plant. In the secondary furnace, additional blended gases with O_2 are added and temperature is increased to decompose the hazardous constituents. The combustion products in the off-gas are then collected in various sorbents in the sampling train.

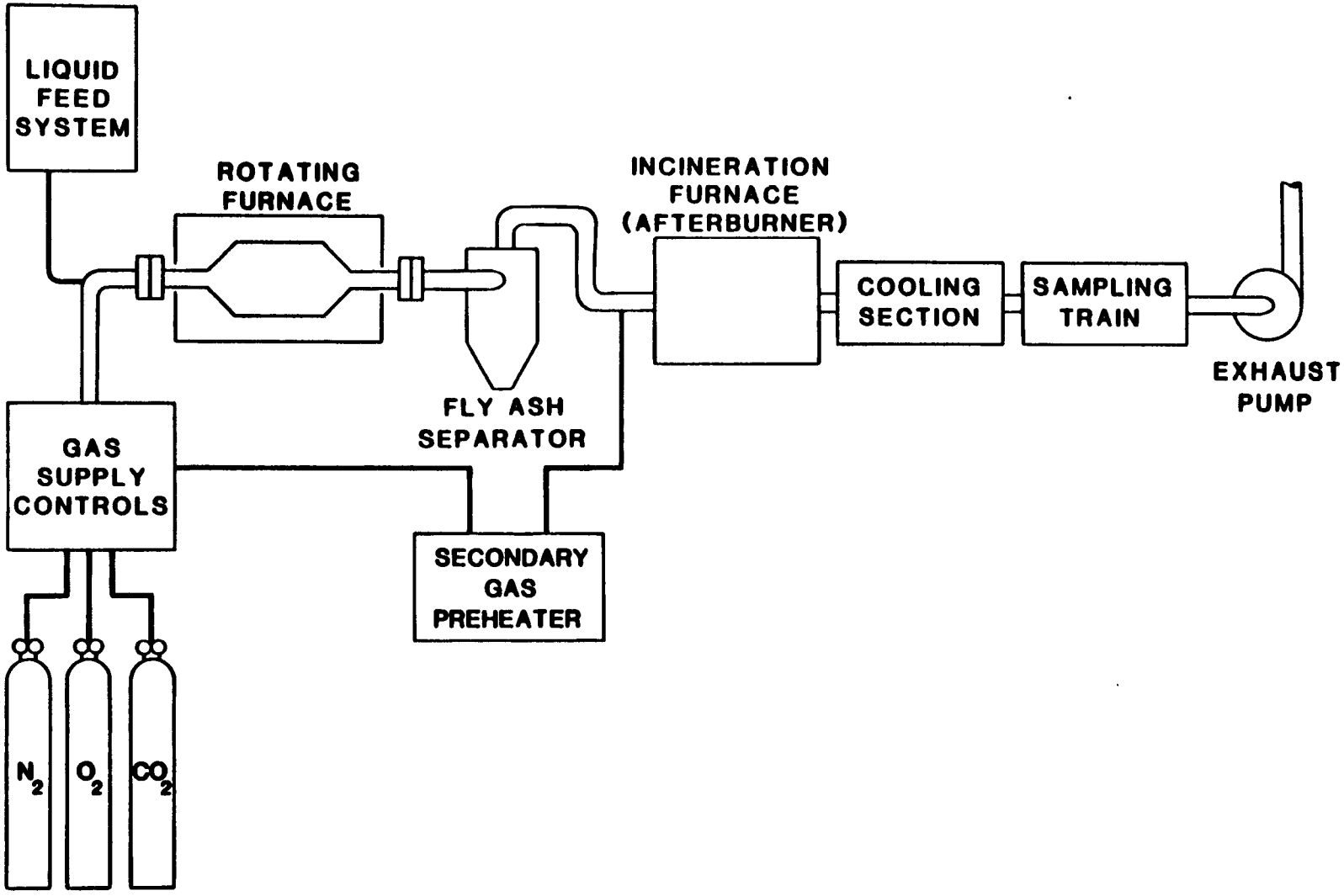


FIGURE 3.2-1

LABORATORY SCALE INCINERATION UNIT

3.2.1 Primary Furnace

The primary furnace (Figure 3.2-2) was an electric furnace with a programmable temperature controller capable of maintaining 1000°C with gas flows up to 20 liters per minute. A gas supply system was used to provide blends of nitrogen (N_2), carbon dioxide (CO_2), and O_2 to simulate various combustion processes in fuels. The primary furnace barrel (Figure 3.2-3) was 130 millimeters (mm) in diameter and 200 mm in length. During test conditions the maximum temperature rise of the primary furnace was about 5.5°C per minute and the carrier gas velocity was maintained between 6 and 8 centimeters (cm) per second.

3.2.2 Fly Ash Trap

A fly ash separator was installed between the primary and secondary furnace. The purpose of this separator was to remove ash from the carrier gas and to prevent plugging of the secondary furnace. The ash separator was a cyclone-type design capable of removing particulates as small as 100 microns. It was constructed of stainless steel and insulated to prevent heat loss between the primary and secondary furnaces.

3.2.3 Secondary Combustion Gas

Additional gases were introduced between the primary and secondary furnaces to simulate secondary combustion gases. The composition of this gas was the same as that of the primary carrier gas which increases the total gas flow rate by 50 percent. The carrier gas was preheated to near ($\pm$ 50°C) that of the primary carrier gas temperature.

3.2.4 Secondary Furnace

The secondary furnace was designed to heat gases up to 1200°C from the primary furnace along with the secondary airflow and to maintain the gases at this temperature for between 2 and 5 seconds. To have fully developed flow while avoiding high pressure losses in the furnace, a velocity range of 20 cm/sec to 500 cm/sec was established. For this velocity range and the

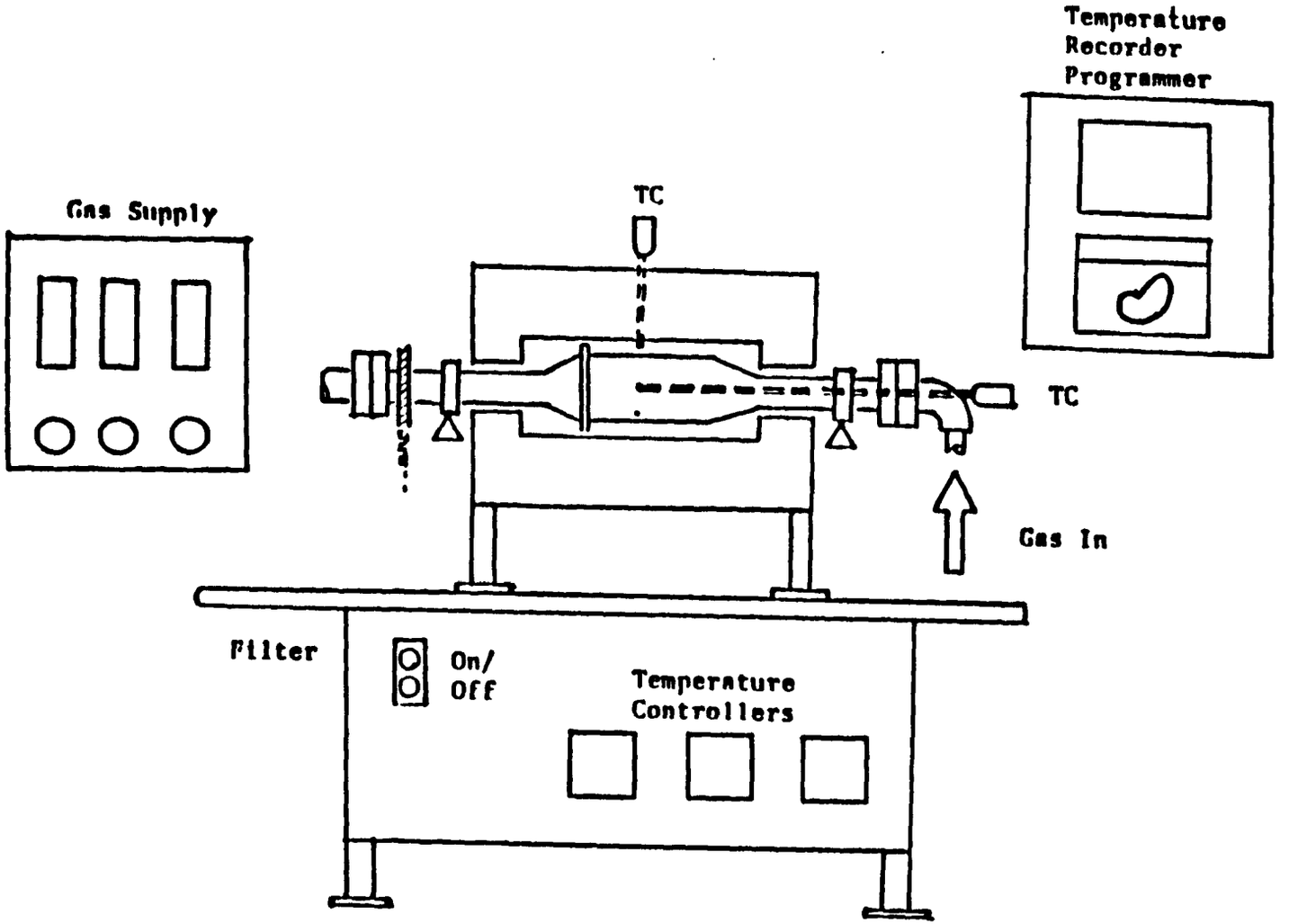


FIGURE 3.2-2
ROTATING TUBE FURNACE ARRANGEMENT

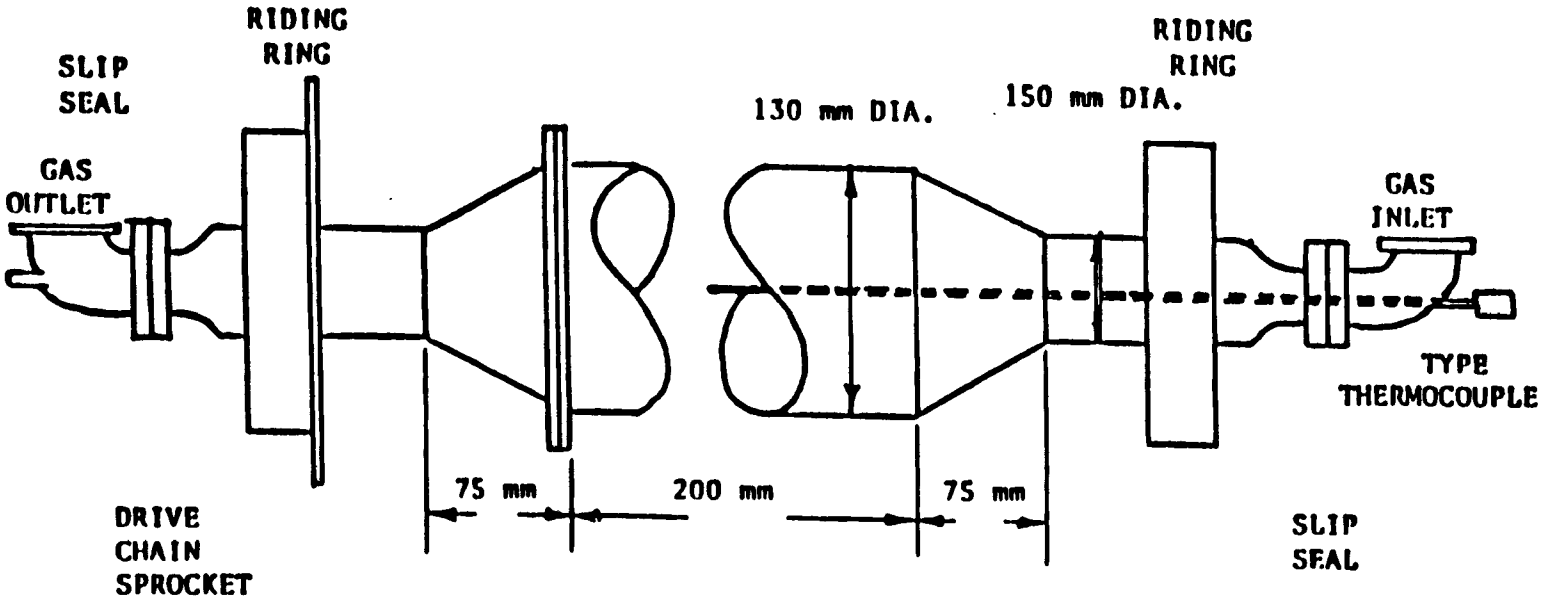


FIGURE 3.2-3
ROTATING TUBE UNIT

desired gas flow rate, the furnace tube diameter was approximately 2-1/2 cm. To maintain a residence time of 5 seconds, the furnace tube was built approximately 10 meters long. The secondary furnace tube was constructed of fused quartz to provide a nonreactive environment at high temperature. Two furnace tube lengths were used to provide 2 and 5 second residence times in the secondary burner.

3.2.5 Cooling Section

The cooling section consisted of a straight 2.5 cm diameter quartz tube approximately 3 feet long. Exit temperature from the cooling section was monitored to insure that temperature was maintained between 200°C and 300°C. Insulation was applied to the tube to adjust the exit temperature.

3.2.6 Sample Collection

All off-gases from the secondary furnace entered the sample collection system that was designed to remove organic and inorganic constituents of concern. A pump was used downstream of the sample collection system to maintain near-atmospheric pressure in the entire flow train. The sample collection system is described in detail in Section 3.3.

3.3 SAMPLING

3.3.1 Introduction

Gases generated from all test incineration runs require a collection system of nonparticulate and particulate fractions. In general, the sampling apparatus for collecting off-gas effluents includes three major components:

- One or more thermostatically controlled compartments to maintain the gas at a temperature consistent with the collection medium, usually hot (200°C) for particulate collection and cool (20°C) for sorbent collection of the more volatile constituents;

- Sample collectors, such as filters and sorbents; and

- o Vacuum pump and gas meter.

The sampling train used is shown in Figure 3.3-1. This device is physically similar to the Modified Method 5 (MM5) sampling train.

3.3.2 Particulate and Residue Collection

Bottom residue left in the kiln from the test burn was removed by the most efficient means available to the lab which was consistent with:

- o Complete removal (>99 percent);
- o Prevention of outside contamination; and
- o Prevention of damage to the kiln.

Residue removal and cleaning of the kiln were made to assure subsequent test burns were not cross-contaminated. Bottom residue was stored at about 4°C in glass bottles with Teflon-lined caps until combined with the fly ash.

The fly ash separator retained the larger particulates carried through the primary furnace tube. The fly ash was removed and stored at about 4°C.

Filter cassettes were used to trap particulates which were not separated as fly ash and may vary in size from 1 to 100 microns. The filter used was a glass fiber-type and was stored at 4°C. in a glass bottle with a Teflon-lined cap.

Figure 3.3-2 illustrates the flow of the residue sample into the analytical system. The three solid fractions from the test burns were weighed and the weight summed to estimate the percentage of sample volatilized:

$$\text{Percent Sample Volatilized} = \left(1 - \frac{W_B + W_F + W_P}{W_S}\right) \times 100$$

Where W_B = Weight of bottom residue
W_F = Weight of fly ash
W_P = Weight of filter particulates
W_S = Weight of original sample

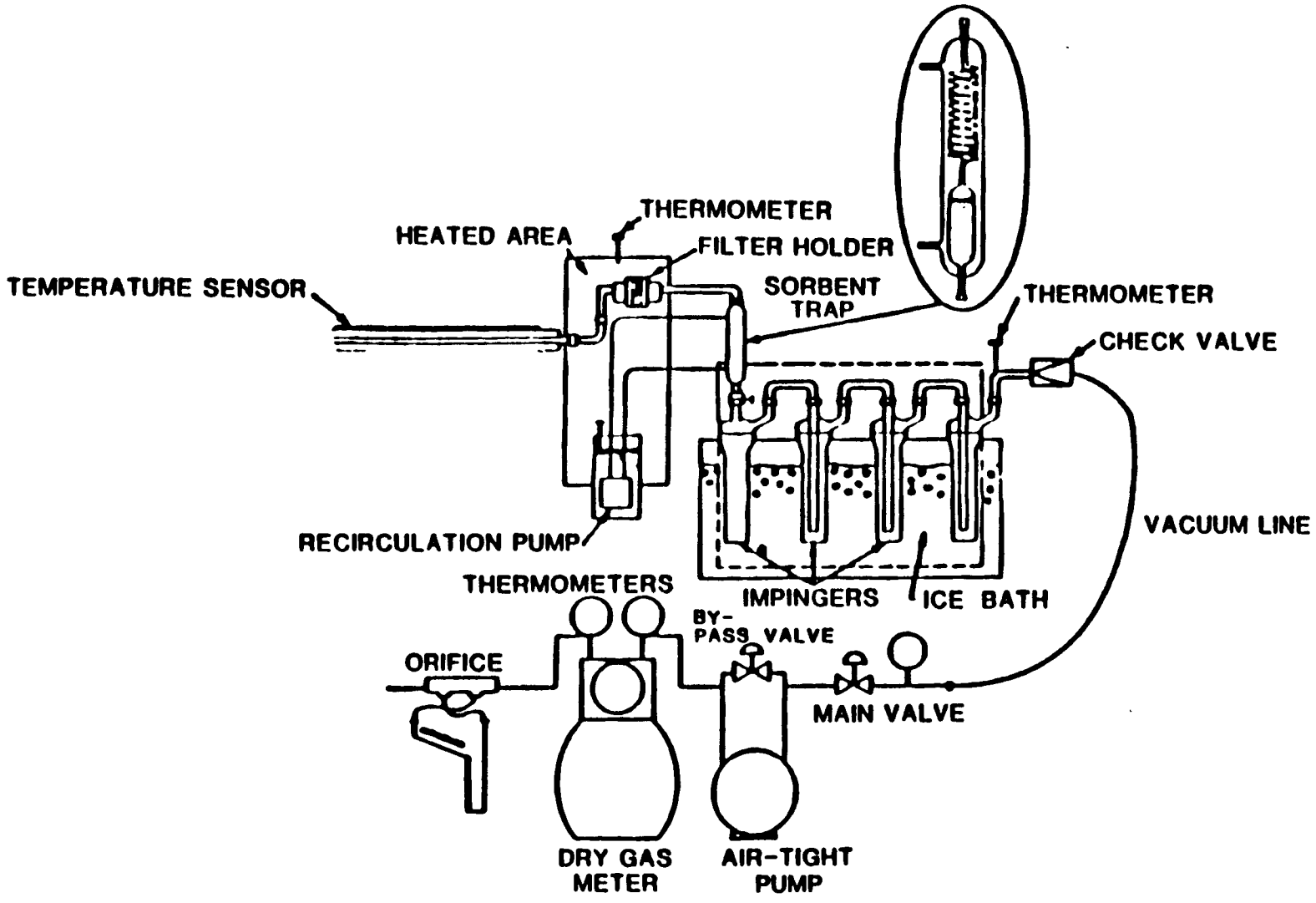


FIGURE 3.3-1
SAMPLE TRAIN

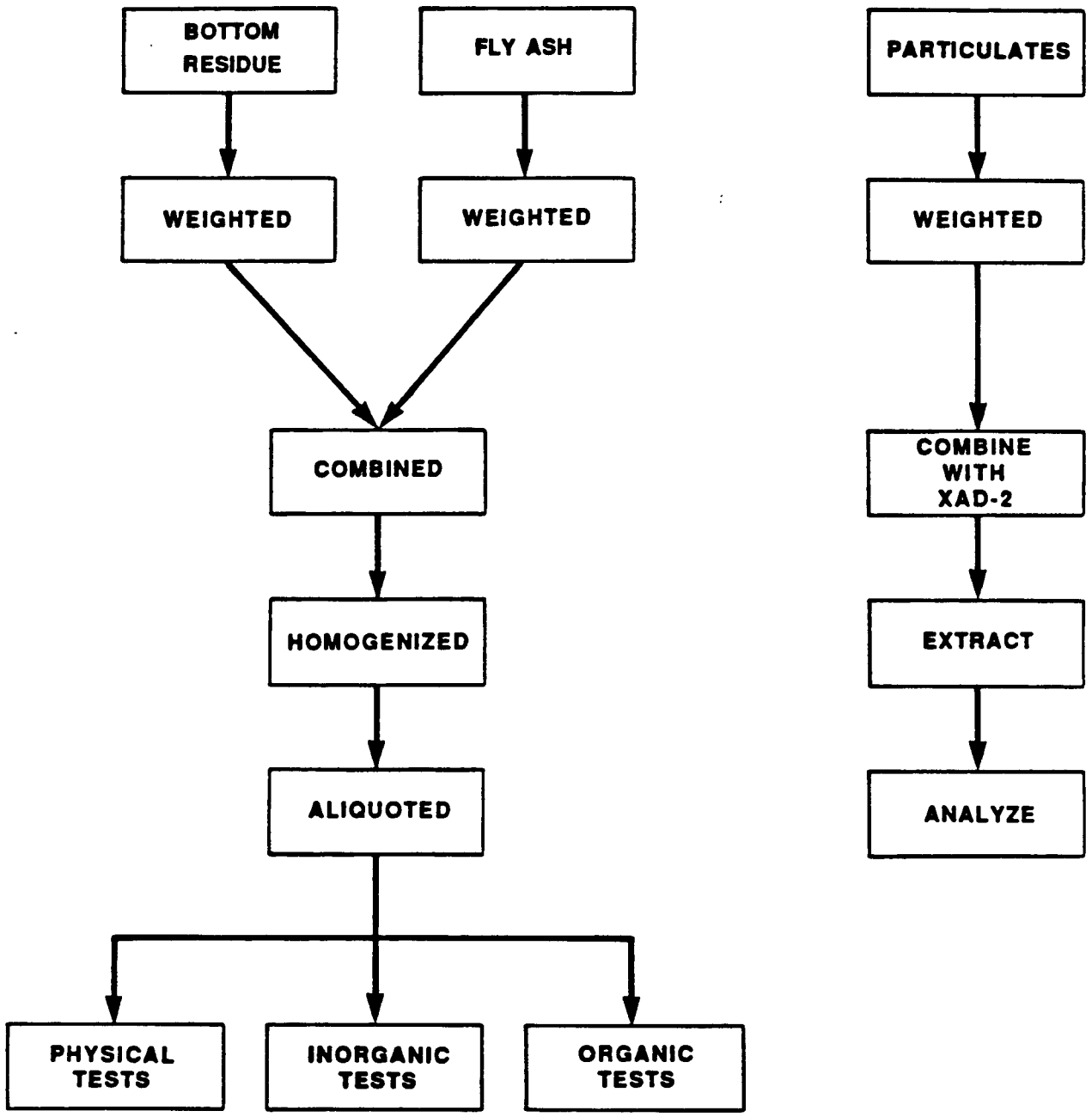


Figure 3.3-2
SOLID RESIDUE COLLECTION FLOW CHART

The bottom ash and fly ash were combined and homogenized. Aliquots of this residue were taken for the various chemical and physical analyses required to determine the distinction efficiency of the POHCs.

The particulate filter was weighed and combined with the XAD-2 resin for extraction and analysis.

Table 3.3-1 describes the types of sorbent and impinger solutions that were used to trap organic and inorganic products from the incineration. Figure 3.3-3 depicts the sampling train.

After a test run, the sorbents and impinger fractions, as well as the condensate when applicable, were transferred to glass bottles with Teflon-lined caps for storage at about 4°C.

3.4 OPERATING PROCEDURES

The following sections outline operational considerations for the soil, sludge, and liquid tests.

3.4.1 Soil Tests

The typical operation sequence for the bench-scale soil sample testing is as follows:

- Weigh out appropriate sample size (200-500 grams ± 0.5 grams).
- Place the sample in the kiln barrel and bolt the barrel halves together.
- Place the kiln barrel into the furnace and attach the thermocouple and gas connections.
- Set the secondary furnace temperature and allow it to reach test condition temperature before proceeding.

TABLE 3.3-1

GAS SAMPLE COLLECTION MATRIX

Compound Class	Sorbent	Impinger	Water Trap*
Volatile Organics	Tenax/Charcoal		Test
Semivolatile Organics	XAD-2		Test
Volatile Metals		Silver Catalyzed Ammonia Persulfate	Test
Acid Compounds		0.1 NaOH	Test
Cyanide		0.1 NaOH	Test
Basic Compounds		0.1 HCl	Test

*A water trap will be utilized when the test sample is sludge or liquid. (See text.)

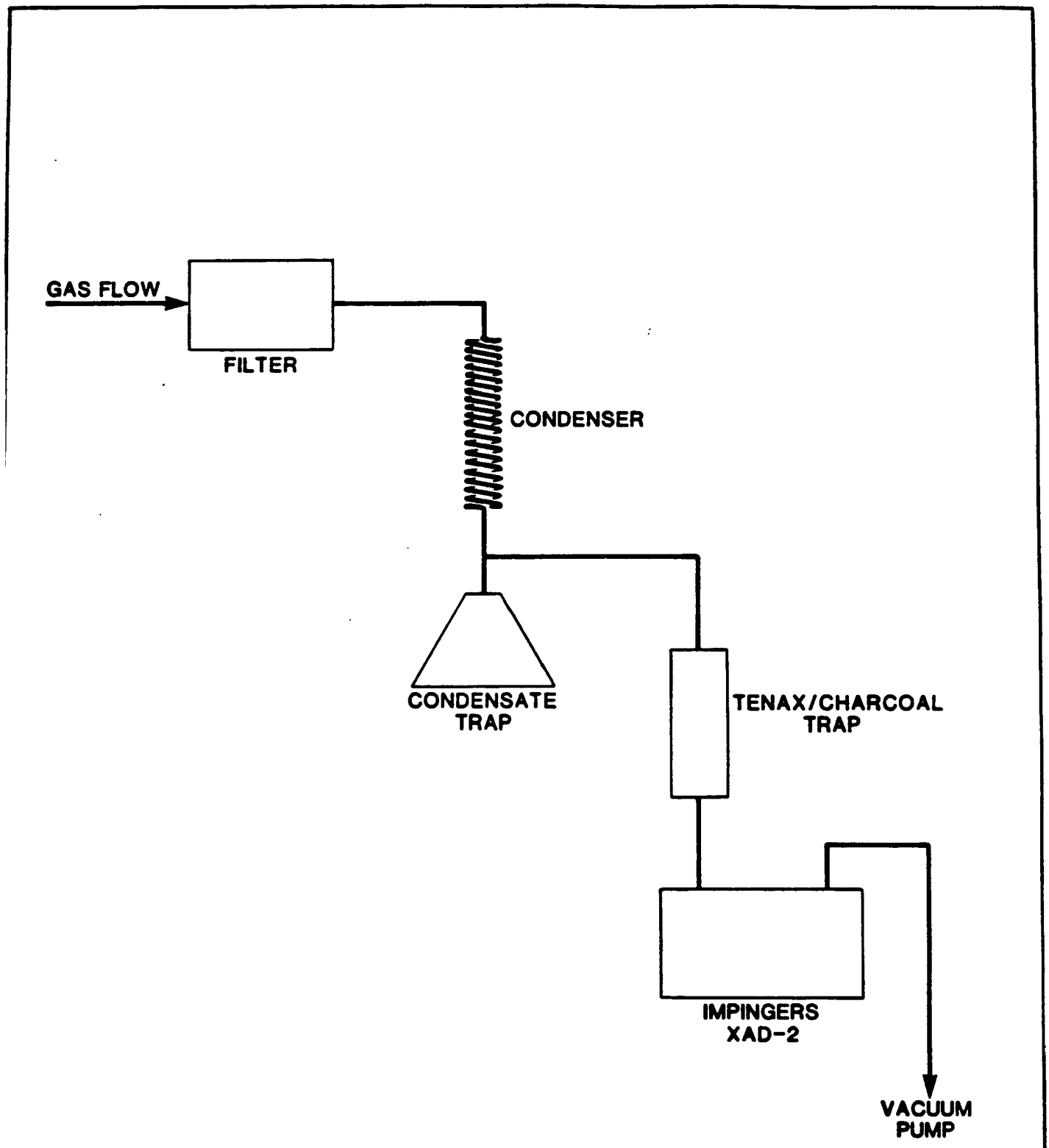


FIGURE 3.3-3
MODIFIED SAMPLING TRAIN
FOR HIGH MOISTURE SAMPLES

- Switch on the evacuation exhaust pump.

- Establish carrier gas flow at the desired blend and flow rate.

- Start temperature ramp on primary furnace.

- After reaching the desired test temperature on the primary furnace, maintain desired test conditions for one hour before starting shutdown procedures.

- Turn primary furnace off and stop barrel rotation, but continue gas flow.

- After primary furnace has cooled to 400°C, turn off secondary furnace.

- Divert gas from sampling train and remove collected samples.

- After primary furnace has cooled to near room temperature, remove kiln barrel and disassemble.

- Remove residual sample from barrel.

- Disassemble fly ash collection system and remove fly ash.

- During the course of the system operation, the following parameters were monitored and recorded: N_2, CO_2, and O_2 flow rates of primary and secondary gasses, temperature of the rotating kiln gas, fly ash separation system exit gas, secondary furnace and cooling section exit gas, particulate sample isothermal box, and impinger isothermal box. The sample train flow meter pressure differential also was monitored.

4.0 BENCH-SCALE INCINERATION TEST CONDITIONS

4.1 SELECTED VARIABLES

The bench-scale test matrix was developed recognizing the typical operating parameters for hazardous waste incinerators capable of handling chemically contaminated solids. These parameters are residence time, temperature, and oxygen level necessary in the combustion process for complete destruction of organics.

4.1.1 Time Parameters

The residence time of the waste materials in the primary burner was selected on the assumption that a full-scale incineration system should be able to vaporize all organics associated with Basin F soils or overburden materials within an hour. Therefore, the residence time for the primary burner was limited to a maximum of one hour operation at the selected operating temperature(s).

The variation in residence time in the secondary burner, however, was based upon values in hazardous waste incineration literature. The minimum value of 2 seconds appears in virtually all scientific and engineering publications concerning hazardous waste incineration, and was consistent with the data presented (Frankel et al., 1983) for commercially operated afterburners.

The 5-second value appears near the upper end of the scale (Bonner et al., 1981).

4.1.2 Temperature Parameters

The temperature parameters in the primary and secondary chambers were selected based on the limitation of the available laboratory equipment and the general operating practices for the incineration of hazardous wastes.

The primary chamber (Linder furnace) used in the bench-scale setup can operate at the maximum temperature of 1000°C. The typical operating ranges for the rotary kiln, fluid bed, and multiple hearth furnace are:

Rotary kiln	820°C - 1600°C
Fluidized bed	450°C - 980°C
Multiple hearth	660°C - 1000°C

Based on the above assumptions, the operating temperatures selected for the primary burner were: 650°C, 800°C, and 900°C. (Note: For safety reasons, the Linder furnace was not operated at the peak value of 1000°C).

The first temperature in the secondary burner at 900°C which is consistent with the minimum afterburner temperature (Frankel et al., 1984) for afterburners associated with rotary kilns.

The maximum temperature in the afterburner, 1,200°C, represented a practical upper limit of the bench-scale equipment. Further, it is a midrange temperature for afterburners as (Frankel et al., 1984).

A third temperature, 650°C, has been chosen as a minimum value for test purposes. This temperature is consistent with the low end of values shown for afterburners. Further, it is at the low end of temperatures where 99.99 percent DRE for hazardous organics is achieved (Dellinger et al., 1984).

The third temperature provided a matrix of six points for the establishment of time and temperature requirements to incinerate the soils. The matrix appears as:

	Temperature		
Time	Minimum	Intermediate	Maximum
2 seconds	650°C	900°C	1,200°C
5 seconds	650°C	900°C	1,200°C

4.1.3 Oxygen Concentration

Oxygen concentration determines the level of excess air that is optimal in firing the supplementary fuel. Oxygen concentration was varied in the carrier gas as a means of making the bench-scale tests most representative of the postflame oxidation regions as well as the pyrolysis region. Oxygen concentration influences not only the temperatures achieved in the flame (see, for example, Babcock and Wilcox, 1978, for a correlation between excess O_2 and flame temperature), but also influences the degree of combustion completeness and the minimization of PIC formation.

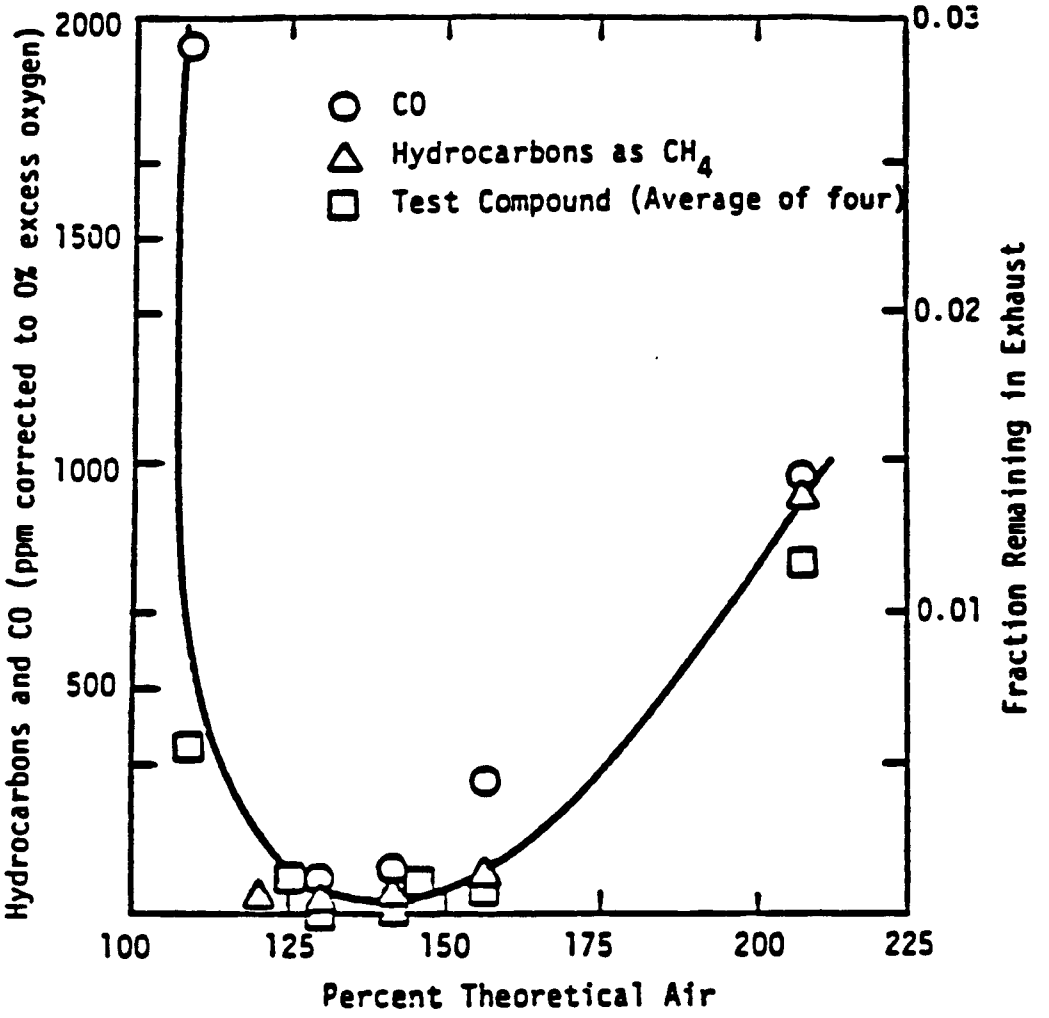
The research previously cited demonstrates (Kramlich et al., 1984) that PICs are minimized and DREs are maximized with excess air in the 30 to 40 percent range. Below and above that range, PICs increase in dramatic quantities, as is shown in Figure 4.1-1.

The minimum concentration of O_2 in the exhaust gas was selected at 5.4 percent, corresponding to the apparent optimal value shown in Figure 4.1-1. This level was set for the carrier gas in the experiment.

The maximum concentration of O_2 in the exhaust gas was set at 7.0 percent, corresponding to common firing practices of many combustion systems. Further, this representation of 50 percent excess air represented a practical upper limit beyond which DRE levels of 99.99 percent could not be expected (see, for example, Figure 4.1-1).

4.2 ANALYTICAL PARAMETERS MONITORED

The analytical parameters monitored to measure the destruction efficiency of each test burn were twenty-two (22) semivolatile organic compounds that are target compounds for all RMA field investigation programs (Ebasco, 1986b). However, only ten semivolatile compounds (Aldrin, Dieldrin, Endrin, Isodrin, DCPD, DBCP, CPMS, CPMSO, $CPMSO_2$ and Supona) were detected in the overburden sample through baseline analyses (see Table 2.4-4). These ten compounds were selected as PHOCs. The analytical method employed for feed sample was



(constant air velocity, variable load, equal molar mixture of chloroform, benzene, chlorobenzene, and acrylonitrile added 3 percent by weight to heptane).

FIGURE 4.1-1

EXHAUST CO AND TOTAL HYDROCARBONS AND FRACTION OF TEST COMPOUND REMAINING IN EXHAUST AS A FUNCTION OF THEORETICAL AIR

gas chromatography-mass spectrometry (GC/MS) at full scan, while for better sensitivity GC/MS Selective Ion Monitoring (SIM) mode was used for residue and off-gas samples.

5.0 TEST RESULTS

5.1 SUMMARY

Initially, 25 test burns were conducted on overburden and surrogate samples. Six test burns were aborted due to equipment malfunction. Three of the test burns were conducted on surrogate compounds to demonstrate the efficiency of the sampling trains. The remaining test burns were successfully completed; Table 5.1-1 presents the test matrix for these 16 successful runs.

Analyses for the 10 selected POHCs indicated complete (more that 99.99 percent) destruction of organics associated with Basin F overburden material at most of the test conditions. Further evaluation of selected runs for PICs resulted in the selection of preliminary optimum combustion conditions for the complete destruction of organics. Two additional test burns of overburden sample were conducted at these optimum conditions and upon evaluation of identified PICs in off-gases from these two optimization test runs, the optimum combustion conditions were selected for a full-scale incineration system concept design.

5.2 FEED SAMPLE ANALYSES

The feed sample (overburden from Basin F) for each test burn was analyzed for 22 semi-volatile organic compounds (target compounds) by GC/MS full scan analytical method as certified by USATHAMA. Appendix A contains the analytical results as reported by the laboratory (Hittman-Ebasco), while Appendix B contains the chemical structure of each of the 22 compounds.

A summary of results of the feed sample analyses is presented in Table 5.2-1. The table identifies parameters that were reported to have concentrations higher than their respective analytical detection limit. The parameters identified in Table 5.2-1 are Aldrin, Dieldrin, Endrin, Isodrin, DCPD, DBCP, CPMSO, $CPMSO_2$ and Supona.

TABLE 5.1-1

TEST MATRIX

Test Run	Temp °C in Primary Burner	Temp °C in Secondary Burner	Detention Time (min) in Primary Burner At Operating Temp.	Detention Time (sec) in Secondary Burner	O_2 Level in Secondary Burner	Type of Feed in Primary Burner	Remarks
2	900	1200	60	2	7%	Soil	Good
3	900	900	60	2	7%	Soil	Good
5	900	1200	60	5	7%	Soil	Good
6	650	650	60	5	7%	Soil	Good
7	900	900	60	5	7%	Soil	Repeat of Run 4; Good
8	650	1200	60	5	5.4%	Soil	Good
9	900	900	60	5	5.4%	Soil	Good
10	900	1200	60	2	5.4%	Soil	Good
11	650	650	60	2	7%	Soil	Good
12	900	900	60	2	5.4%	Soil	Good
13	900	1200	60	2	5.4%	Soil	Good
14	650	650	60	2	5.4%	Soil	Good
17	650	650	30	2	5.4%	Soil	Good
18	650	900	60	2	5.4%	Soil	Good
19	800	900	30	5	7%	Soil	Test conditions for a fluidized bed
20	800	900	15	2	7%	Soil	Test conditions for a fluidized bed

TABLE 5.2-1

FEED SAMPLE (ug/gm)

Test Run	Aldrin	Dieldrin	Endrin	Isodrin	DCPD	DBCP	CPMS	CPMSO	$CPMSO_2$	Supona
1	Aborted									
2	2200	1800	310	240	160	41	2000	120	490	13
3	2100	1600	350	300	170	47	1900	140	460	14
5	1700	1200	240	200	120	33	1500	95	360	13
6	2400	1600	310	110	150	36	2300	51	200	19
7	2300	1800	330	130	110	31	2100	57	200	19
8	1900	1400	260	100	110	28	2100	47	170	15
9	2300	1800	340	130	110	29	2100	47	160	22
10	1700	1000	170	89	59	13	1400	31	150	7.8
11	3600	1800	300	190	100	23	2100	53	270	17
12	3700	1800	320	180	93	22	2000	53	250	16
13	2300	1100	200	110	70	15	1600	46	190	11
14	3600	1600	390	220	88	20	1700	84	310	20
17	3100	1500	370	180	85	12	2100	100	330	21
18	3500	1600	400	190	140	42	2600	99	330	22
19	3900	1800	500	210	160	48	2700	110	360	26
20	3700	2800	610	180	240	49	2600	91	280	34

As can be seen from this table, the samples used for test burns were not homogeneous. The concentration of Aldrin ranged from 1700 to 3900 ppm while Dieldrin ranged from 1000 to 2800 ppm. Among Endrin, Isodrin, and DCPD, the concentrations ranged from 170 to 610 ppm, 89 to 300 ppm, and 59 to 240 ppm, respectively. Among the chlorophenylmethyl sulfur compounds, CPMS had the highest concentration for each test burn from 1400 to 2700 ppm. Concentrations of DBCP and Supona were found in the range of 7.8 to 49 ppm, respectively.

5.3 RESIDUE ANALYSES

Residue remaining after each test burn was analyzed for all target organic compounds to determine the completeness of organic volatilization from the feed samples. The GC/MS-SIM mode was employed for analyses of organic compounds. Results of analyses of residue samples, as reported by the laboratory, are presented in Appendix A.

A summary of results of these analyses is presented in Table 5.3-1. Table 5.3-1 shows those 10 parameters that were detected in feed samples, i.e., Aldrin, Dieldrin, Endrin, Isodrin, DCPD, DBCP, CPMS, CPMSO, $CPMSO_2$ and Supona. As can be seen from this table, almost all organics associated with the overburden samples were volatilized under test conditions (650°C - 900°C).

5.4 OFF-GAS ANALYSES

Off-gases from the combustion process was collected in the sampling train. Off-gas samples comprised condensates, accumulated materials on the filter, and materials absorbed on carbon and XAD-2 resins. The analytical method employed for off-gas samples was GC/MS-SIM. Results of these analyses as reported by the laboratory, are shown in Appendix A. A summary of results of analyses of off-gas samples, identifying ten principal organic compounds (i.e., Aldrin, Dieldrin, Endrin, Isodrin, DCPD, DBCP, CPMSO, $CPMSO_2$, and Supona) is presented in Table 5.4-1 and Appendix A.

TABLE 5.3-1

RESIDUE ANALYSES (ug/gm)

Test Run	Aldrin	Dieldrin	Endrin	Isodrin	DCPD	DBCP	CPMS	CPMSO	$CPMSO_2$	Supona
2	<.05	0.15	<0.08	<0.08	<0.008	<0.008	<0.08	<0.08	0.25	<0.08
3	<.05	<0.02	<0.08	<0.08	<0.008	<0.02	<0.08	<0.08	<0.08	<0.08
5	<.03	0.20	<0.05	<0.01	33	<0.005	<0.05	<0.05	<0.05	<0.05
6	<.03	<0.01	<0.05	<0.05	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
7	0.03	<0.01	<0.05	<0.05	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
8	0.18	0.10	<0.05	<0.01	<0.005	<0.005	<0.05	0.06	<0.05	<0.05
9	0.05	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
10	0.23	0.10	<0.05	<0.01	<0.005	<0.005	0.08	<0.05	0.05	<0.05
11	3.0	2.8	<0.05	0.04	<0.005	<0.005	<0.05	<0.05	4.0	<0.05
12	0.05	0.03	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	0.87	<0.05
13	<0.03	<0.02	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
14	0.07	0.04	<0.05	<0.01	<0.005	<0.005	1.1	<0.05	0.10	<0.05
17	0.25	0.06	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
18	0.81	0.93	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
19	<0.03	<0.02	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
20	<0.03	<0.02	<0.05	<0.01	<0.005	<0.005	0.06	<0.05	<0.05	<0.05

TABLE 5.4-1

CONTAMINANTS REMAINING IN OFF-GASES (ug)

Test Run	Aldrin	Dieldrin	Endrin	Isodrin	DCPD	DBCP	CPMS	CPMSO	$CPMSO_2$	Supona
2	21	23	<0.05	0.50	<0.005	1.1	27	<0.05	3.2	<0.05
3	480	190	<0.05	8.8	<0.005	<0.005	57	290	<0.05	<0.05
5	<0.03	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
6	<0.03	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
7	0.14	0.11	<0.05	<0.01	<0.005	<0.005	0.13	<0.05	<0.05	<0.05
8	<0.03	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
9	<0.03	25	<0.05	0.85	<0.005	<0.005	6.44	<0.05	1.94	<0.05
10	<0.03	0.36	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
11	<0.03	3.5	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	37
12	<0.03	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
13	<0.03	<0.01	<0.05	<0.01	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
14	0.07	1.2	<0.05	0.39	<0.005	<0.005	<0.05	<0.05	1.4	18
17	2.0	<0.01	<0.05	0.39	<0.005	<0.005	<0.05	<0.05	<0.05	<0.05
18	0.49	0.25	0.20	<0.01	<0.005	1.8	3.8	<0.05	<0.05	<0.05
19	0.30	0.94	3.7	<0.01	<0.005	<0.005	<0.05	<0.05	<0.15	<0.05
20	<0.03	0.55	4.6	<0.01	<0.005	<0.005	2.0	<0.05	12	<0.05

As can be seen from this table, the organic species remaining in the off-gas samples were below the analytical detection limits most of the time. It can be concluded that the original organic compounds present in the feed sample can be transformed into some other species if not completely oxidized to CO, CO_2, and H_2O in the specified test conditions.

5.5 DETERMINATION OF DRE

The Resource Conservation and Recovery Act regulation designates the destruction and removal efficiency (DRE) of principal organic hazardous constituents (POHC) as the requirement for incinerator design (Federal Register, 1981). The DRE of an incinerator system is defined as:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} \times 100$$

where DRE = destruction and removal efficiency, %

W_{in} = mass feed rate of the principal organic hazardous constituent(s) to the incinerator

W_{out} = mass emission rate of the principal organic hazardous constituent(s) to the atmosphere (as measured in the stack prior to discharge).

Thus, destruction and removal efficiency calculations are based on the combined efficiencies of destruction in the incinerator and removal from the gas stream in the air pollution control system. The potential presence of principal organic hazardous constituents in incinerator bottom ash or solid/liquid discharges from air pollution control devices is not accounted for in the destruction and removal efficiency calculation as currently defined by regulations.

The regulations require a DRE of 99.99 percent for all principal organic hazardous constituents of a waste, unless it can be demonstrated that a higher or lower destruction and removal efficiency is more appropriate based on human health criteria.

Based on the concept described above, the DRE of each of the 10 organic compounds found in the feed sample was determined. The mass feed rate of each organic compound was determined by multiplying concentration times the mass of feed sample used in each test burn. The total weight in micrograms of each organic species present in each feed sample, as reported by the laboratory, is included in Appendix A.

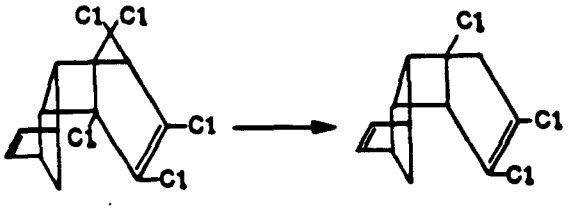
Table 5.5-1 presents the DRE of 10 principal hazardous organic constituents at all test conditions. As can be seen from this table, a DRE of more than 99.99 percent was achieved for each POHC at most of the test conditions.

5.6 ANALYSIS OF COMBUSTION RESULTS

To understand the observed thermal decomposition or stability of organic species detected in feed samples, a discussion on the expected thermal stability of detected organic species is presented below.

Aldrin

Aldrin is a bridged chlorinated hydrocarbon. This molecule can undergo a very low energy concerted four-center elimination of hydrogen chloride (HCl) (see rxn 1).



(rxn 1)

The resulting olefin will have strained bonds at the site of HCl elimination and be expected to undergo further decomposition. Four center concerted eliminations of HCl have activation energies (E_a) on the order of 45-50 kcal/mole and frequency factors (A) of $10^{13.5}$ - 10^{14} s^{-1} (Benson, 1976). This means that >99.99 percent destruction efficiency is expected for temperatures around 600-650°C at 2.0 seconds gas phase residence time ($\bar{t}_r$). Since the reaction is unimolecular, the rate will not depend on the oxygen concentration or the concentration of any other component of the waste feed.

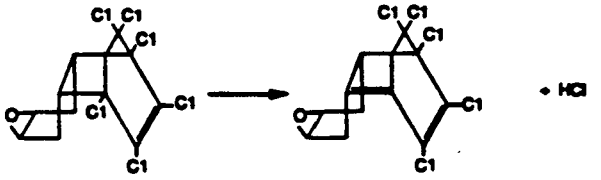
TABLE 5.5-1

DESTRUCTION AND REMOVAL EFFICIENCY OF TEN PRINCIPAL HAZARDOUS ORGANIC CONSTITUENTS IN OVERBURDEN SAMPLE

Temp Degrees C in Secondary Burner	650			900							1200			
Temp Degrees C in Primary Burner	650			650	800		900				650	900		
Gas Residence Time in Second Burner (In Seconds)	2		5	2	2	5	2		5		5	2		5
Oxygen Level in Off-Gas (%)	5.4	7	7	5.4	7	7	5.4	7	5.4	7	5.4	5.4	7	7
Run Number	14 17	11	6	18	20	19	12	3	9	7	8	10 13	2	5
ALDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.94	100.00	100.00	100.00	100.00 100.00	100.00	100.00
CPMS	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00 100.00	100.00	100.00
CPMSO	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.41	100.00	100.00	100.00	100.00 100.00	100.00	100.00
$CPMSO_2$	100.00 100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.00
DBCP	100.00 100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	99.99	100.00
DCPD	99.99 100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.00
DIELDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.97	100.00	100.00	100.00	100.00 100.00	100.00	100.00
ENDRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.00
ISODRIN	100.00 100.00	100.00	100.00	100.00	100.00	100.00	100.00	99.99	100.00	100.00	100.00	100.00 100.00	100.00	100.00
SUPONA	99.74 100.00	99.38	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00 100.00	100.00	100.00

Dieldrin

This molecule can also undergo a low energy elimination of HCl (rxn 2). In addition, the epoxide linking is weak and will undergo homolysis at low



(rxn 2)

temperatures. This molecule is also expected to be destroyed in the vicinity of T = 650°C and $\bar{t}_r$ = 2.0 s.

Endrin

Endrin is a geometric isomer of Dieldrin and is expected to undergo the same reactions.

Isodrin

Isodrin is also expected to undergo HCl elimination and decompose under conditions similar to Aldrin, Dieldrin, and Endrin.

DCPD (Dicyclopentadiene)

Dicyclopentadiene is a bicyclic hydrocarbon. Since it does not contain any chlorine, it cannot undergo any low energy HCl eliminations as was the case for the previously discussed pesticides. However, many of the bonds in the molecule are stressed and would be expected to break under mild thermal exposure, as a consequence it is not surprising that this molecule can be destroyed at relatively low temperatures, probably below 750°C and 2.0 seconds residence time.

$CPMSO_2$ (Chlorophenylmethylsulfone)

Benzyl methyl sulfone has been studied and observed to undergo homolysis at the carbon sulfur bond with an A factor of $10^{14.52}$ s^{-1} and E_a = 51.2

kcal/mole (Benson & O'Neal, 1970). $CPMSO_2$ could undergo the same bond homolysis although the carbon-sulfur bond would be much stronger in $CPMSO_2$.

Attack by radical species (hydroxyl [OH] in an oxidative environment or hydrogen [H] in a pyrolytic environment) may be the primary mode of destruction through abstraction of a H on the methyl substituent. The authors are not aware of kinetic or mechanistic studies of attack on the sulfone group itself. Destruction at temperature below 800°C and 2.0 s residence time seem reasonable.

CPMSO (Chlorophenylmethylsulfoxide)

The authors are not aware of directly relevant studies in the literature. The same general comments made for $CPMSO_2$ would apply to CPMSO.

DBCP (Dibromochloropropane)

This molecule may undergo a four center elimination of HCl and hydrogen bromide (HBr). Eliminations of HBr are an even lower energy pathway than HCl (E_a = 40-45 kcal/mole (Benson, 1976). Consequently, this molecule is expected to be very unstable forming several possible inhalogenated olefins at temperatures below 600°C.

CPMS (p-Chlorophenylmethylsulfone)

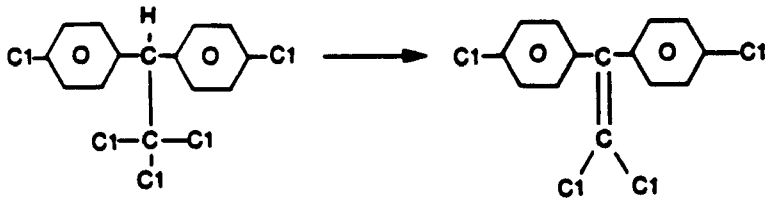
The sulfur linkage in this molecule is isoelectronic with oxygen; consequently, its behavior under thermal stressing is expected to be similar to that of an ether linkage. There are no low energy concerted pathways or weak bonds which would readily break upon heating. Electrophilic addition of OH to the ring or abstraction of H from the methyl group are the most likely pathways of destruction. This compound may be moderately stable although it should not be particularly difficult to destroy by controlled incineration.

Supona

Supona is a relatively complex molecule with a number of functional groups that can decompose by various mechanisms. The dichlorophenyl group would be expected to be quite stable. The phosphate ester may undergo a 6 center elimination in analogy to those observed for normal esters (Benson & O'Neal, 1970). The latter type of reaction can be quite rapid ($E_a \approx 47$ kcal/mole and $A \approx 10^{12}$ s^{-1}), resulting in the formation of the carboxylic acid and an olefin. Bond homolysis may also occur at the carbon-oxygen linkage in Supona. As a result Supona may not be very stable, decomposing below 750°C and 2.0 seconds residence time.

Other Low Level Chlorinated Pesticides (Chlordane, DDT, DDE, HCCPD)

Chlordane and DDT can undergo low energy elimination of HCl. DDE is the elimination product of DDT and should be considerably more stable (rxn 3).



(rxn 3)

DDE will be degraded by radical attack and probably require temperatures in excess of 750°C at 2.0 s residence time.

HCCPD is a perchlorinated molecule and consequently cannot undergo HCl elimination. However, the five membered ring is strained and would be expected to break at relatively low temperatures. It should be noted that this molecule may react to form the very stable molecule, hexachlorobenzene, in significant yields.

Other Low Level Phosphonated Pesticides (Malathion and Parathion)

Parathion may decompose by loss of the nitro group at temperatures of around 700°C and residence time of 2.0 s. Malathion will likely undergo a 6 center

elimination at the ester functional group to form the corresponding acid and ethylene. The Malathion decomposition may occur at less than 700°C.

Other Low Level Contaminants (DIMP, DMMP, Vapona, Atrazine, Oxathiane, Dithiane)

With the exception of Atrazine, each of these molecules is a phosphonate. DIMP may undergo a 6 center elimination at low temperatures; however, the pathway is not possible in DMMP and Vapona. Consequently, the latter two compounds may be more stable, although still not extremely stable.

In Atrazine, the amine groups may be susceptible to radical attack at intermediate temperatures through hydrogen abstraction or radical addition followed by elimination of the amine. Atrazine may be expected to decompose between 700 and 800°C at 2.0 seconds residence time.

Oxathiane and Dithiane are isoelectronic. Neither molecule contains any weak bonds which would readily break under thermal stressing. Abstraction of H by OH is the most likely mode of destruction. Both species are expected to be fairly stable, although they should represent no special problems for the incineration of Basin F wastes.

5.7 ANALYSIS OF PRODUCTS OF INCOMPLETE COMBUSTION

Evaluation of initial results of test burns indicated that the DRE of 99.99 percent for organic compounds could be achieved at all test conditions. Since the objective of multiple test burns is to determine the optimum combustion conditions for complete destruction of organics present in the feed sample, the following assumptions were postulated for further evaluation.

- Organic compounds present in the feed sample can be destroyed to 99.99 percent at test conditions;

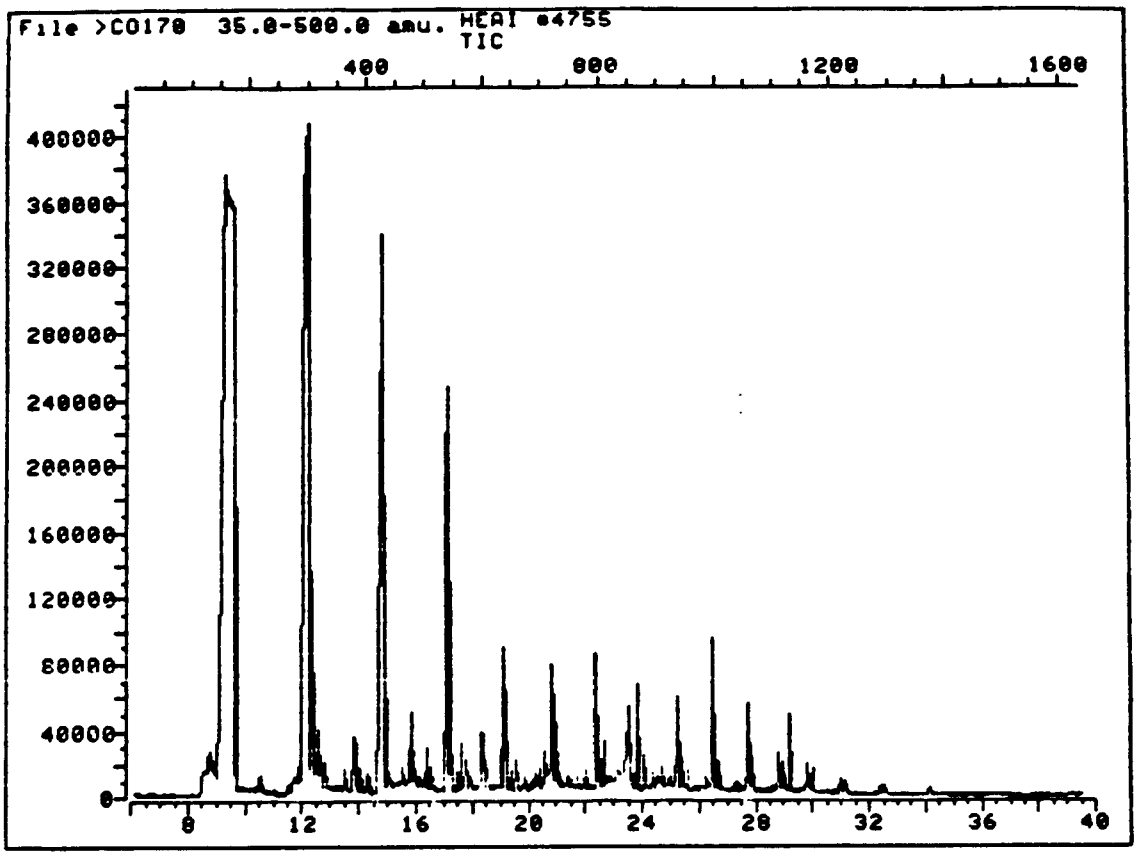
- Test burns performed with a 2-second residence time in the secondary burner would most likely fail to destroy organics at 99.99 percent level; and

- The most complete destruction of organics should produce minimum numbers and quantities of toxic PICs. Therefore, the test burn that produced the least number and quantities of toxic PICs in the off-gas sample should be selected as the run with the optimum combustion conditions.

Based on these postulations, the off-gas samples from Runs 12, 13, and 14 were selected for the analyses of PICs. GC/MS-SIM mode was used for PICs analyses (see Figures 5.7-1 to 5.7-3). The results of these analyses are presented in Tables 5.7-1 to 5.7-3. The chemical structures of the identified PICs are provided in Appendix C. The compounds (PICs) identified were screened for toxicity characteristics. Some compounds were identified as toxic compounds (irrespective of dose or concentration) in accordance with the Registry of Toxic Effects of Chemical Substances. The greatest number and yield were observed from Run No. 14 with the primary and secondary burners at only 650°C, while the least number and yield were observed for Run No. 13 with the primary and secondary burners at 900°C and 1200°C, respectively. The resulting toxic PICs for all three runs are summarized in Table 5.7-4. To understand pathways of PICs, a general discussion of the mechanism of formation of each class of compound is presented. Specific compounds are discussed when they are of particular interest. Non-toxic products are not addressed.

Aliphatics and Substituted Aliphatics

Although not typically reported in combustion studies, simple straight chain and cyclic aliphatics (e.g., hexane and methylcyclopentane) may be formed by a variety of radical molecule interactions involving smaller hydrocarbons. They may also result from the thermal decomposition of higher molecular weight hydrocarbons. The observed alcohols, carbonyls, and esters are typical partial oxidation products for hydrocarbons for temperatures below 450°C. It is suspected that the observed oxidation products may be forming in cool regions of the transfer line in the laboratory combustor. However, the authors cannot absolutely rule out the possibility that these products are formed in the "cool" soil and escape destruction in the gas phase.



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data file header from : ›C0170

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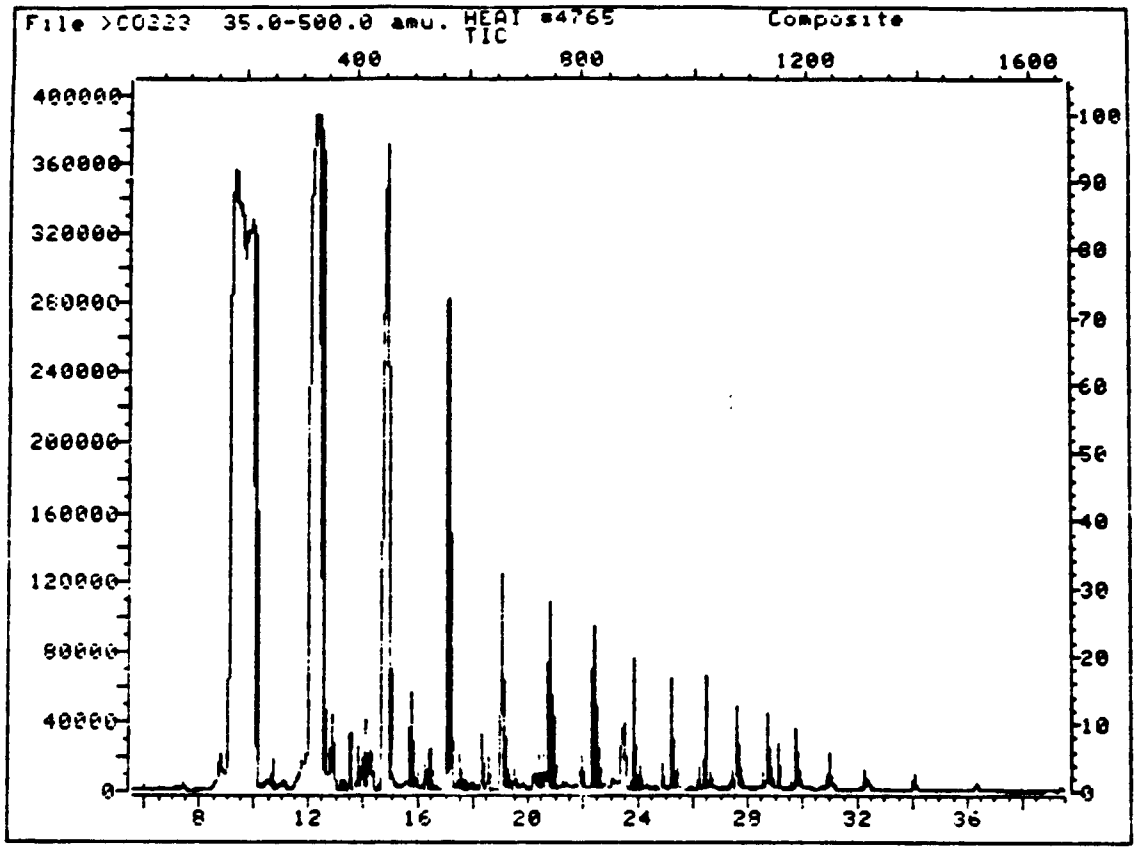
ɔc : BTL# 2
ɔ. #: 2 MS model: 96 SW/HW rev.: 1A ALS # : 0
1ethod file: HE9501 Tuning file: MT9501 No. of extra records: 1
ɔurce temp.: 190 Analyzer temp.: 200 Transfer line temp. : 250

Chromatographic temperatures :	45.	300.	0.	0.	0.
Chromatographic times, min. :	4.0	10.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	10.0	0.0	0.0	0.0	0.0

Figure 5.7-1
RECONSTRUCTED ION CHROMATOGRAPH FOR PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES OF RUN NO. 12

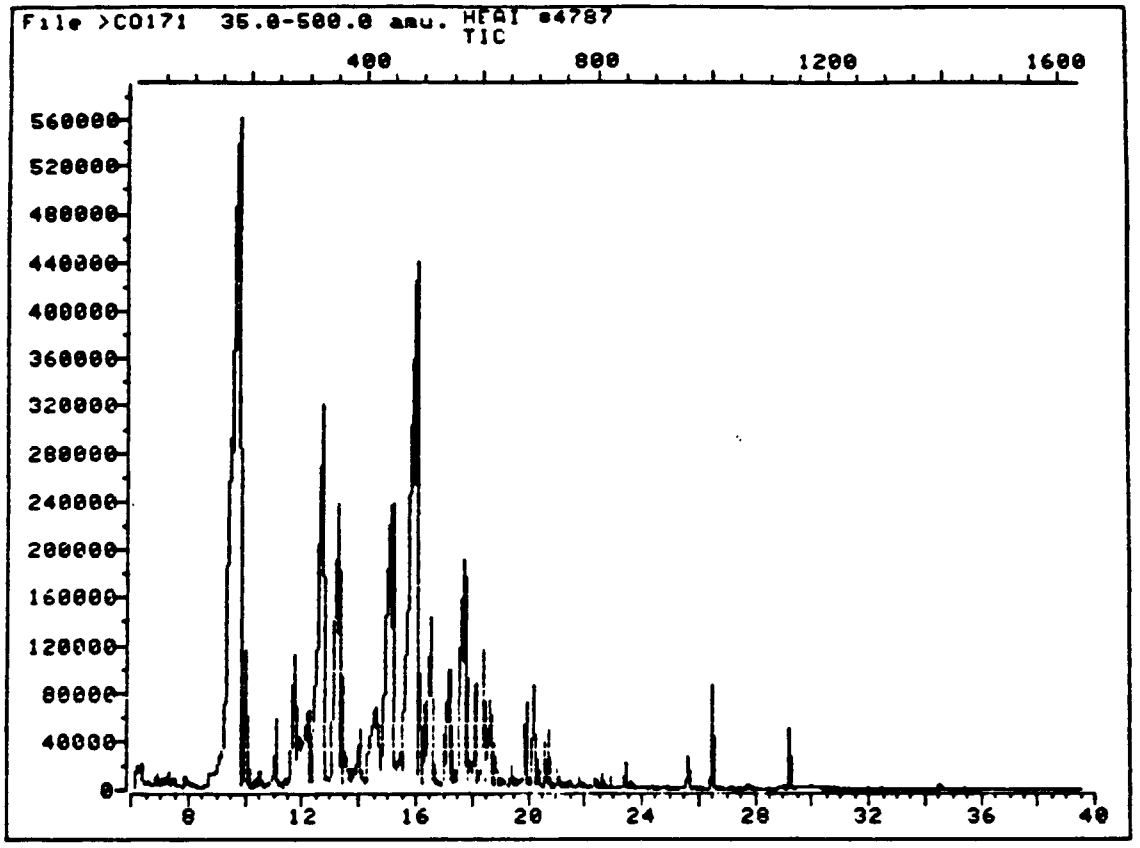


data file header from : >CO223

nple: HEAI #4765 Operator: CHUCK MS 10/27/86 1:54
sc : Composite BTL# 2
s. #: 2 MS model: 96 SW/HW rev.: IA ALS # : 0
Method file: HE9501 Tuning file: MT9501 No. of extra records: 1
ource temp.: 190 Analyzer temp.: 200 Transfer line temp. : 250

Chromatographic temperatures :	45.	300.	0.	0.	0.
Chromatographic times, min. :	4.0	10.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	10.0	0.0	0.0	0.0	0.0

Figure 5.7-2
RECONSTRUCTED ION CHROMATOGRAPH FOR PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES OF RUN NO. 13



data file header from : >CO171

mple: HEAI #4787 Operator: CHUCK MS 10/19/86 21:39
sc : BTL# 3
s. #: 2 MS model: 96 SW/HW rev.: IA ALS # : 0
Method file: HE9501 Tuning file: MT9501 No. of extra records: 1
ource temp.: 190 Analyzer temp.: 200 Transfer line temp. : 250

Chromatographic temperatures :	45.	300.	0.	0.	0.
Chromatographic times, min. :	4.0	10.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	10.0	0.0	0.0	0.0	0.0

Figure 5.7-3
RECONSTRUCTED ION CHROMATOGRAPH FOR PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES OF RUN NO. 14

TABLE 5.7-1

LIBRARY SEARCH RESULTS OF PRODUCTS OF INCOMPLETE COMBUSTION
IN OFF-GAS SAMPLE FROM RUN NO. 12

Compound	Estimated Total Amount (ug)
Cyclohexane	1500
Methyl Cyclopentane	620
3-Methyl-2-Butanone	160
Benzene	1200
Hexane	3800
2,2-Dimethyl Hexane	420
Chlorobenzene	250
Hexamethyl Cyclotrisiloxane	4700
Octamethyl-Cyclotetrasiloxane	5000
Decamethyl-Cyclopentasiloxane	4500
Unknown (scan #306 voa) Primary m/z 285	330
Naphthalene	170
Dodecamethyl-Cyclohexasiloxane	1800
Unknown (scan #430 voa) Primary m/z 73	280
2-Pentadecyl-1, 3-Dioxolane	720
Unknown (scan #632 voa) Primary m/z 73	190
o,o,o-Tris-Trimethyl epinephrine	160
Unknown (scan #717 voa) Primary m/z 73	210
Unknown (scan #795 voa) Primary m/z 73	180
Sulfur, mol. (S8)	430
Hexanedioic acid, dioctyl ester	240

TABLE 5.7-2

LIBRARY SEARCH RESULTS OF PRODUCTS OF INCOMPLETE COMBUSTION
IN OFF-GAS SAMPLE FROM RUN NO. 13

Compound	Estimated Total Amount (ug)
2-Methyl Benzofuran	470
Octamethyl Cyclotetrasiloxane	13000
Unknown (scan #301) Primary m/z 73	160
Decamethyl Cyclopentasiloxane	12000
Unknown (scan #339) Primary m/z 285	2200
Unknown (scan #355) Primary m/z 293	160
Unknown (scan #388) Primary m/z 73	170
Unknown (scan #401) Primary m/z 73	130
Unknown (scan #412) Primary m/z 327	170
Unknown (scan #423) Primary m/z 73	240
Dodecamethyl-Cyclohexasiloxane	6500
12-methyl Tetradecanol	1200
Unknown (scan #496) Primary m/z 73	260
12-methyl-1-Tetradecanol	1900
Unknown (scan #569) Primary m/z 64	230
N-methyl-5-nitro-2-Pyridinamine	260
12-methyl-1-Tetradecanol	700
O,O,O-Tris Trimethylsilyl Epinephrine	360
Unknown (scan #742 voa) Primary m/z 73	540
3,4-bis[(Trimethylsilyl) oxyl]-Estratrienone	210
Unknown (scan #820) Primary m/z 73	480
Sulfur, mol. (S8)	510
Unknown (scan #891) Primary m/z 73	400
Unknown (scan #957) Primary m/z 73	360
Silicate anion tetramer	330
Silicate anion tetramer	260
Unknown (scan #1131) Primary m/z 73	220
Unknown (scan #1182) Primary m/z 73	180
3,5-bis (1,1-dimethylethyl)-1,2-Benzenediol	140

TABLE 5.7-3

LIBRARY SEARCH RESULTS OF PRODUCTS OF INCOMPLETE COMBUSTION
IN OFF-GAS SAMPLE FROM RUN NO. 14

Compound	Estimated Total Amount (ug)
Cyclohexane	1000
3-Chloro-2-Propenenitrile	780
Benzene	1600
Hexane	2800
Unknown (scan #158 voa) Primary m/z 93	700
Unknown (scan #168 voa) Primary m/z 86	2200
Tetrachloroethene	5000
Methylbenzene	940
Chlorobenzene	4300
N-ethyl-Cyclohexanamine	900
Bromobenzene	570
Hexachloro-1, 3-Butadiene	1700
1,4-Dichlorobenzene	6600
1,2-Dichlorobenzene	3500
Octamethyl Cyclotetrasiloxane	850
1-Bromo-2-Chlorobenzene	2400
4-Chloro-Benzonitrile	6500
1,3,5-Trichlorobenzene	8100
1,2,3-Trichlorobenzene	3300
2,6-Dichloro Benzonitrile	940
1,2,4,5-Tetrachlorobenzene	6000
2,4,6-Trichloro Benzenamine	240
5-Bromo-6-methyl-3-(1-methylpropyl)-Pyrimidinedione	250
2,5-Dichloro-Thiazolopyrimidine	790
5,7-Dichloro-Thiazolopyrimidine	560
Pentachlorobenzene	1500
4,7-Dichloro-benzo-2,1,3-Thiadiazole	220
3-Chloro-1,1'-Biphenyl-4-01	370
Unknown (scan #611 semi-vol) Primary m/z 241	300
Hexachlorobenzene	220
Hexachlorodifluoro-Pentadiene	270

TABLE 5.7-4

SUMMARY OF OBSERVED TOXIC PRODUCTS
OF INCOMPLETE COMBUSTION

Compound	Total Amount (ug)		
T Primary (°C)	650	900	900
T Secondary (°C)	650	900	1200
Run No.	14	12	13
Aliphatics and Substituted Aliphatics			
Hexane	2800	3800	ND
Cyclohexane	1000	1500	ND
Methyl cyclopentane	ND	620	ND
2,2 Dimethyl hexane	ND	420	ND
Hexanedioc acid, dioctyl ester	ND	240	ND
12-Methyl-1-tetradecanol	ND	ND	700
3 Methyl-2-butanone	ND	160	ND
Olefins			
Tetrachloroethene	5000	ND	ND
Hexachloro-1,3-butadiene	1700	ND	ND
Aromatics and Substituted Aromatics			
Benzene	1600	1200	ND
Toluene	2200	ND	ND
Benzonitrile	1700	ND	ND
3,5-bis (1,1-dimethyl ethyl)-1,2 benzenediol	ND	ND	140
2-Methyl benzofuran	ND	ND	470

TABLE 5.7-4
(Continued)

Compound	Total Amount (ug)		
T Primary (°C)	650	900	900
T Secondary (°C)	650	900	1200
Run No.	14	12	13
Halogenated Aromatics			
Chlorobenzene	4300	420	ND
1,2 Dichlorobenzene	3500	ND	ND
1,4 Dichlorobenzene	6600	ND	ND
1,3,5 Trichlorobenzene	8100	ND	ND
1,2,4,5 Tetrachlorobenzene	6000	ND	ND
Pentachlorobenzene	1500	ND	ND
Hexachlorobenzene	220	ND	ND
3-chloro-1,1' biphenyl-4-ol	370	ND	ND
4-chlorobenzonitrile	6500	ND	ND
2,6-Dichlorobenzonitrile	940	ND	ND
Heterocyclics			
N-Methyl-5 nitro-2-Pyridinamine	ND	ND	260
5-Bromo-6-methyl-3-(1-methylpropyl) pyrimidinedione	250	ND	ND
Polynuclear Aromatics			
Naphthalene	ND	170	ND
Other			
Sulfur, Mol. (S_8)	ND	430	510

In either case, none of these compounds are on the EPA's Appendix VIII list and represent little cause for practical concern.

Olefins

Tetrachloroethene (PERC) and hexachloro-1,3-butadiene (HCB) were observed in the 640°C run (Run No. 14). These results are not surprising, as these compounds have been observed in a number of other laboratory studies (Taylor & Dellinger; Graham, et al., 1986) Both PICs are expected to be quite stable, especially PERC. HCB can easily be formed from the fragmentation of Aldrin, Endrin, Dieldrin, Isodrin, or Supona. PERC could be formed from the fragmentation of essentially any of the chlorinated pesticides in the feed. Both compounds have also been observed to be formed from radical molecule reactions involving chlorinated C_1 and C_2 species (see rxn for example) (Taylor & Dellinger; Frenklack et al., 1983).

$$\cdot CCl_3 \text{ (or } \cdot CHCl_2) + CHCl_3 \text{ (or } CCl_4) \rightarrow C_2HCl_5 + \cdot Cl \qquad \text{(rxn 4a)}$$

$$C_2HCl_5 \rightarrow \underline{C_2Cl_4} + HCl \qquad \text{(rxn 4b)}$$

$$C_2Cl_3 + C_2Cl_4 \rightarrow \underline{C_4Cl_6} + Cl \qquad \text{(rxn 4c)}$$

The suspected stability of PERC suggests it could be one of the more significant emissions of chlorinated compound from the incineration of Basin F wastes.

Aromatics and Substituted Aromatics

Benzene and toluene (as well as other substituted benzenes such as ethylbenzene and styrene) are frequently observed major PICs, resulting from the combustion or pyrolysis of most organics. They may be formed from the dechlorination and loss of other functional groups from the ring structures of most of the chlorinated pesticides in the Basin F waste. However, it is more likely that they are formed by radical molecule addition and disproportionation reactions similar to those shown in rxn 4 (without the chlorine

substituents) with an additional reaction involving acetylene or methylacetylene (see rxn 5).

$$\cdot C_4H_5 + C_2H_2 \rightarrow \underline{C_6H_6} + H \qquad \text{(rxn 5a)}$$

$$\cdot C_4H_5 + C_3H_4 \rightarrow \underline{C_7H_8} + \cdot H \qquad \text{(rxn 5b)}$$

Benzonitrile can be formed by addition of the nitrile radical to benzene, the nitrile radical resulting from any number of fragmentation reactions of nitrogen containing material in the waste feed. Atmospheric nitrogen is not expected to play a role as temperatures are not high enough to result in degradation of N_2.

The benzedediol would seem to be the result of a low temperature reaction either in a transfer line (or the soil). 2-Methyl benzofuran, on the other hand, may result from either high temperature gas phase reactions or, at lower temperatures, via pathways similar to that proposed for alcohols and carboxylic acid. Benzofuran and methyl benzofuran have been observed in significant yields from other high temperature flow reactor oxidations of organic waste materials (Graham et al., 1986).

Halogenated Aromatics

A variety of halogenated benzenes were observed for the low temperature run (Run No. 14). This is not surprising since chlorinated aromatics have been observed from pyrolysis and oxidation of a number of chlorinated organics (Frenklack, et al., 1986; Graham, et al., 1986). In fact, the presence of chlorine has been shown to effectively increase the yield of aromatic products and chlorinated aromatics (Taylor & Dellinger).

The lower chlorinated aromatics may be a result of bond homolysis of more complex chlorinated species (e.g., CPMSO monochlorobenzene); however, it is more likely that they form by a complex series of radical molecule reactions similar to that already illustrated for the formation of benzene. Chlorine atom addition reactions to already chlorinated aromatic structures may also contribute to the formation of the higher chlorinated aromatics.

The formation of chlorinated benzonitriles may proceed through nitrile radical attack on chlorobenzenes or chlorine atom attack on benzonitrile. The observation of a chlorobiphenyl-ol is puzzling since the chlorinated biphenyl is expected to be very resistant to radical attack and partial oxidation (to form the phenol) without further fragmentation to smaller species.

The formation of chlorobenzenes is important because they have been shown to be very stable, especially under pyrolytic conditions, and may be one of the most difficult PICs (or POHCs) to destroy (Graham et al., 1986; Dellinger et al., 1984).

Heterocyclics

Two nitrogen containing heterocyclics were observed, a substituted pyridine and an pyrimidinedione. The formation of the latter species is puzzling for two reasons. First of all, it would not appear to be very stable and is a very complex species to be formed requiring a very complex series of reactions. The observed pyrimidinedione also has a nitro substituent which is expected to be easily fragmented. This molecule may have been formed in the transfer line after the secondary chamber. In any case, it should be readily destroyed in a full-scale incinerator.

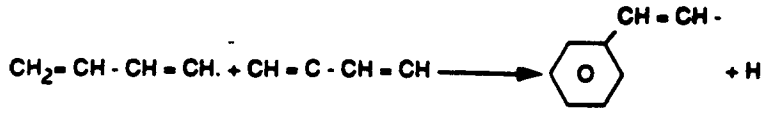
Pyridine is a relatively stable aromatic, expected to be slightly more stable than benzene (Dellinger et al., 1984). No possible direct precursors were identified in the waste feed. Consequently, it is felt that this molecule was formed through a radical molecule addition reaction involving possibly HCN and butadienyl radical in a manner analogous to that shown for the formation of benzene (rxn 6).

$$\cdot C_4H_5 + HCN \rightarrow \underline{C_5H_5N} + \cdot H \qquad \text{(rxn 6)}$$

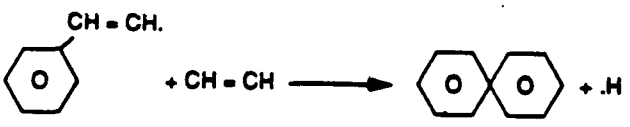
Polynuclear Aromatics

A polynuclear aromatic hydrocarbon such as naphthalene may be formed as an extension of mechanism responsible for benzene. A possible mechanism would

be through a styrene intermediate formed from the reaction of butadienyl radical and vinyl acetylene radical (see rxn 7).



(rxn 7a)



(rxn 7b)

It is not difficult to envision a continuation of this process (involving another vinyl acetylene instead of acetylene) to form higher molecular weight PNAs.

In fact, it is surprising that other PNAs were not reported. Other laboratory studies have shown that PNAs can be the major PIC at higher temperatures even under oxidative conditions (Graham, et al., 1986) The observation of chlorinated benzenes without the observation of chlorinated PNAs is also quite puzzling. In fact, as already discussed, chlorine atoms are implicated in catalyzing the formation of aromatics, PNAs, and soot due to their ability to abstract hydrogen atoms and initiate the reaction sequences shown in reactions 3, 4, 5, and 6 (Taylor & Dellinger; Frenklack, et al., 1983).

Other Emissions

Elemental sulfur (S_8) was observed under all of the conditions tested. These species have also been observed from the thermal degradation of sulfur-containing materials (Taylor & Dellinger). Elemental sulfur is apparently stable enough to resist oxidation at the temperatures and oxygen levels studied. The authors are not aware of any concern by EPA over its emission.

5.8 OPTIMUM COMBUSTION CONDITIONS

Conditions at which Run No. 13 was conducted have been chosen as the optimum combustion conditions for achieving DRE of 99.99 percent for Basin F contaminated soils. Run No. 13 was conducted at the following conditions:

Temperature in the Primary Burner	900°C
Temperature in the Secondary Burner	1200°C
Gas Residence Time in the Secondary Burner	2 seconds
Oxygen Level in Off-Gases	5.4 percent

Reasons for selecting the above conditions as the optimum combustion conditions are discussed in previous subsections. Primarily, these operating conditions are chosen because in Run No. 13 DRE of 99.99 percent for all detected organic compounds and least numbers and quantities of toxic PICs were observed amongst the test runs that were considered to represent the failure (i.e., not achieving appropriate DRE) conditions.

Furthermore, the measurements of the carbon monoxide (CO) concentration levels in the off-gases indicated the most complete combustion of organic contaminants. The concentration of CO in the incinerator off-gases is an indicator for PIC and POHC emissions (Lee & Huffman, 1984). That is, if significant CO emissions are not present, the presence of other carbon-based pollutants would be highly unlikely. Conversely, the presence of significant levels of CO in the combustion products would indicate that the conditions in the incinerator are improper and may result in POHC and other PIC emissions.

Figures 5.8-1, 5.8-2 and 5.8-3 represent the CO levels observed in off-gases for Run Nos. 12, 13, and 14. The graphs were plotted CO levels in off-gases versus temperatures in the primary burner. The least amount of CO was observed during Run No. 13 test burn period.

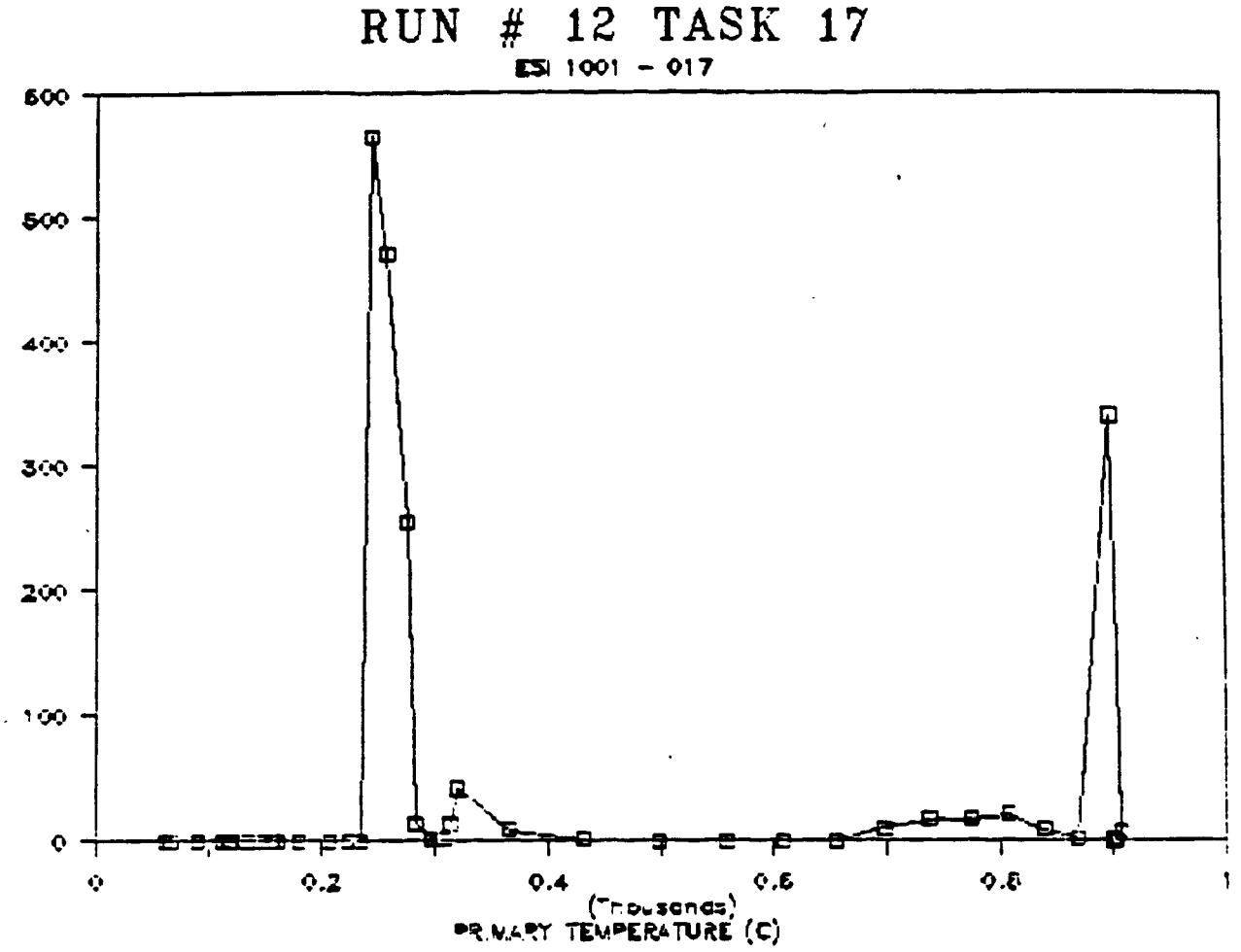


Figure 5.8-1
CONCENTRATION OF CO IN OFF-GASES OF RUN NO. 12

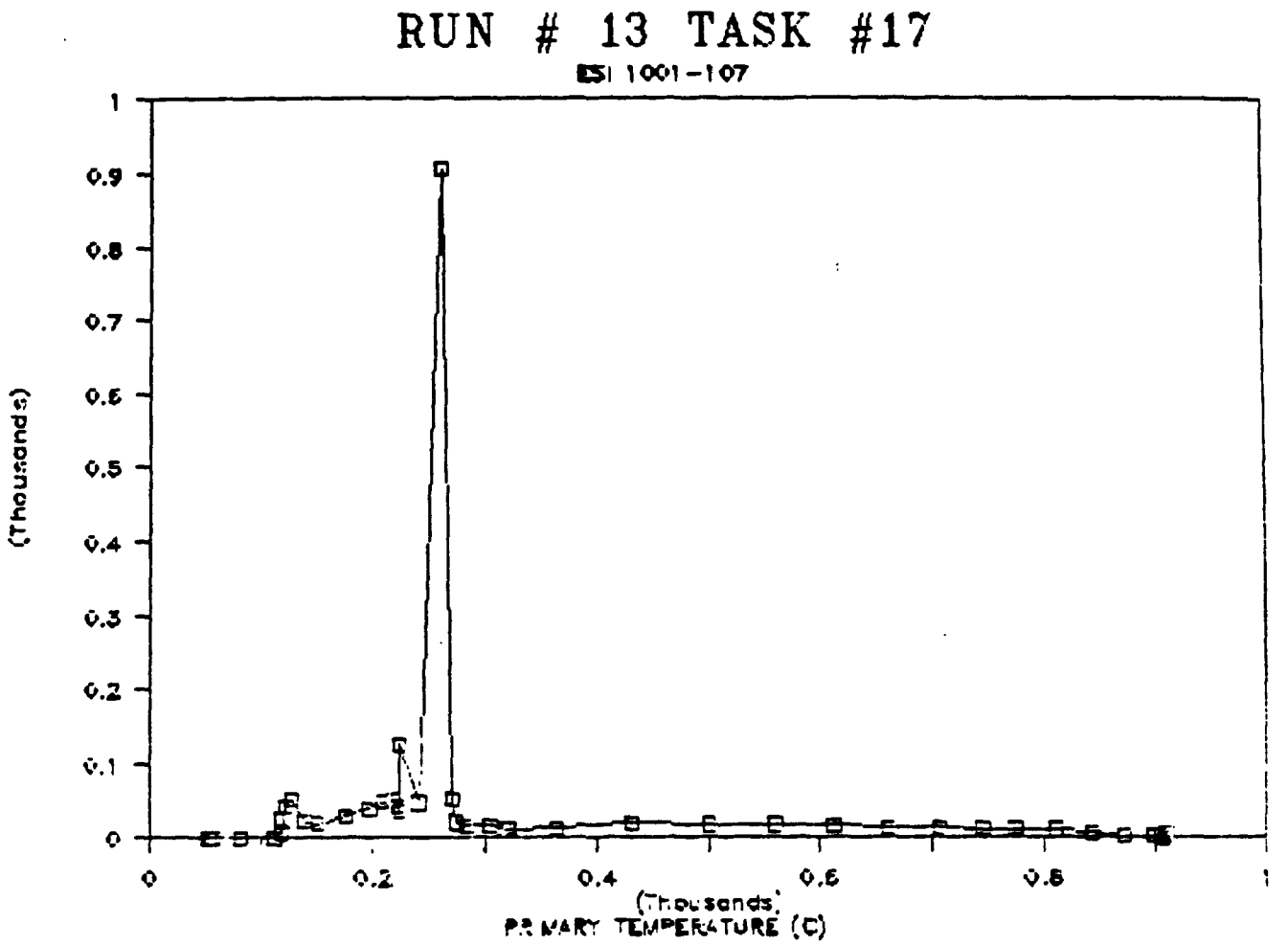


Figure 5.8-2
CONCENTRATION OF CO IN OFF-GASES OF RUN NO. 13

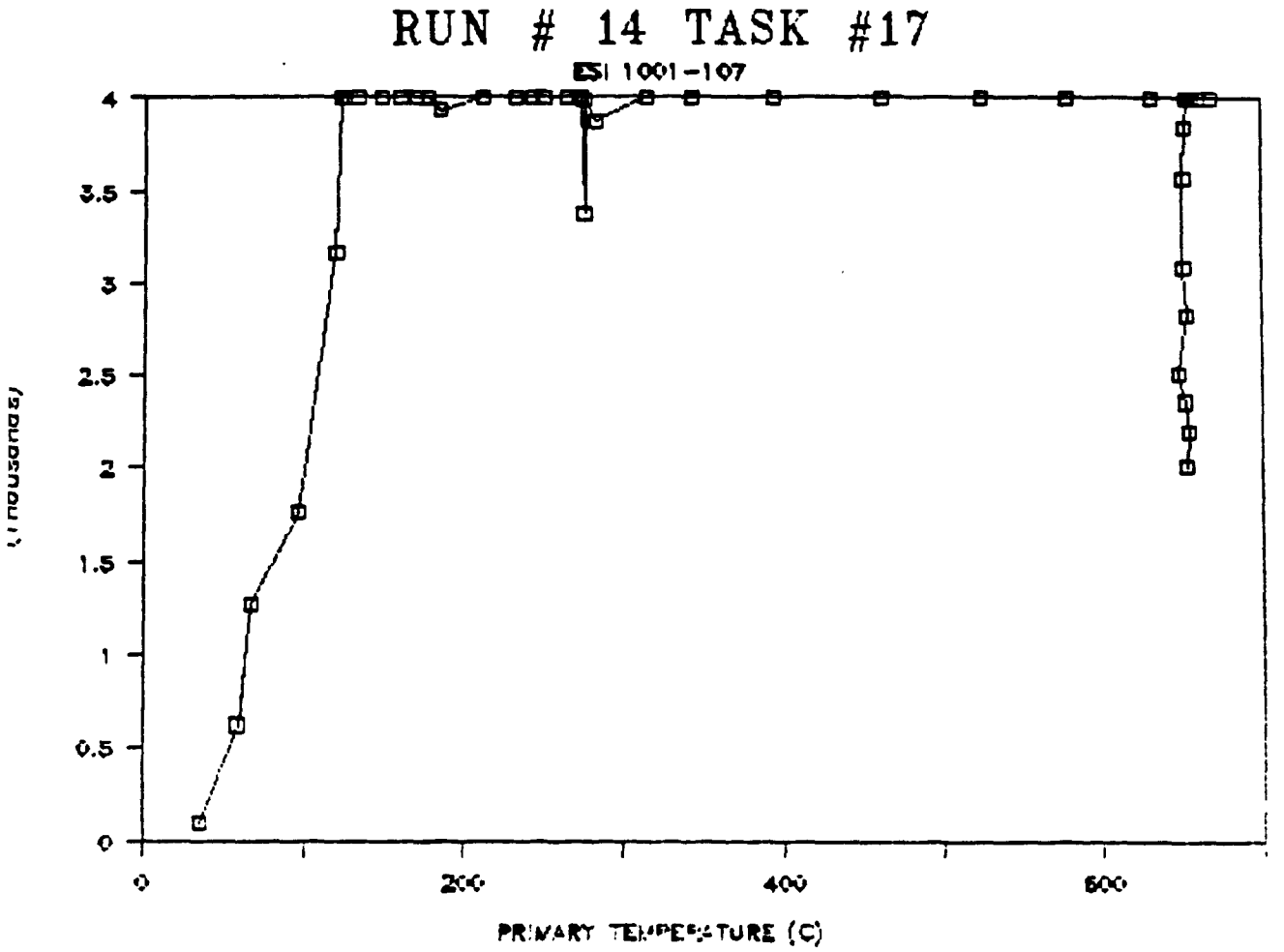


Figure 5.8-3
CONCENTRATION OF CO IN OFF-GASES OF RUN NO. 14

5.9 OPTIMIZATION RUNS

Two additional test burns were conducted at these optimum conditions, with the exception of Run No. 2 which utilized 50 percent excess air (7 percent O_2 in the off-gases) for the purpose of determining the effect of excess oxygen on the formation of products of incomplete combustion. It was found in the literature that the yield and stability of PICs increase with decreased oxygen concentration (Graham, et al., 1986). Figures 5.9-1 and 5.9-2 depict reconstructed ion chromatograms of observed PICs in off-gases of the optimization test runs. Table 5.9-1 presents the comparative evaluation of these two PIC analyses. It can be seen from this table that the test run with 7 percent oxygen level produced the least numbers and quantities of products of incomplete combustion. Off-gas samples were not analyzed for the target organic compounds.

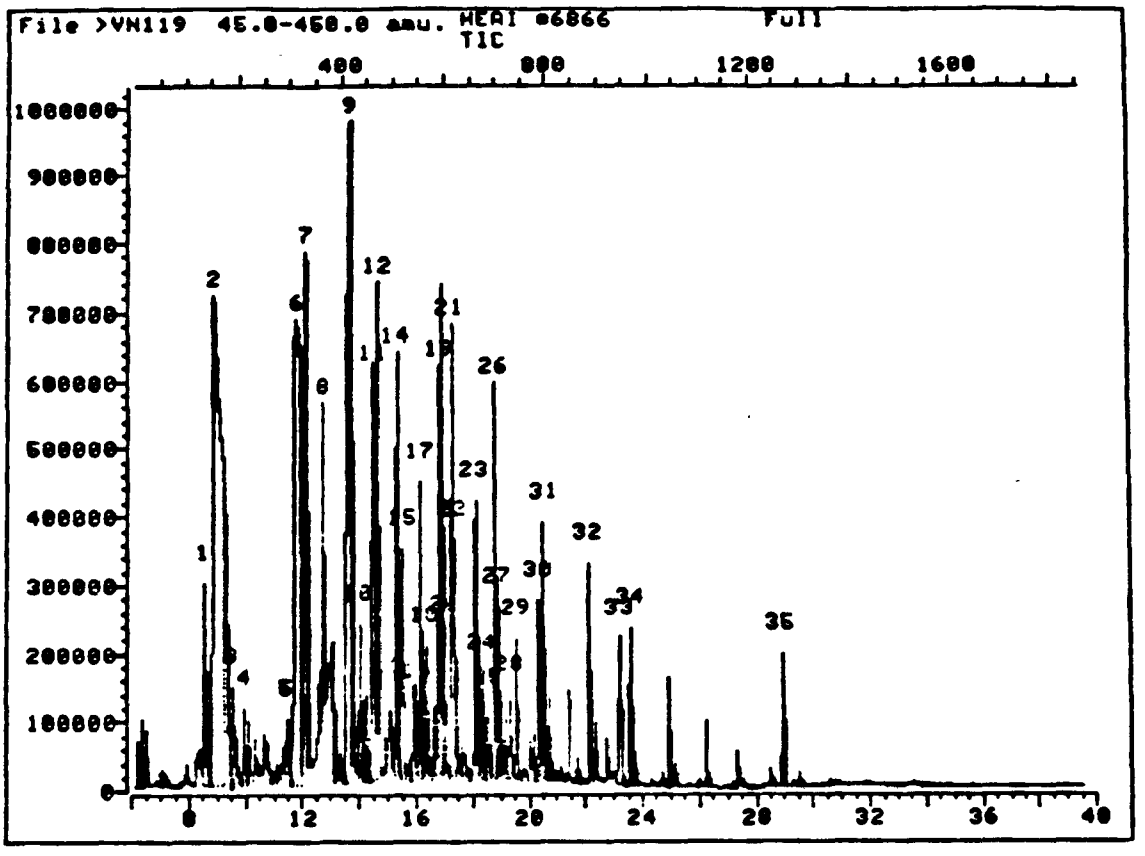
5.10 EP TOXICITY OF RESIDUE

EP toxicity tests were performed on the feed and residue samples of the optimization Run No. 1. The results of the toxicity tests are indicated on Table 5.10-1. No organic parameters in EP leachate were analyzed because the residue samples from all test burns consistently showed target organic compounds below the analytical detection limits. Moreover, for organic analyses, residue samples were extracted using methylene chloride solution. Therefore, it is assumed that no organics can be detected in the EP extract. The EP toxicity test on the feed sample was performed to determine the mobility of toxic metals present in Basin F soils and any effects the incineration process would have on the behavior of these toxic metals.

It can be seen from Table 5.10-1 that the toxic metal concentration in neither extract exceeded the EP Limit concentrations. Moreover, it was observed that arsenic was not reported to be present in the feed extract while small amount of arsenic leached out from the residue sample.

5.11 LIQUID TEST BURN RESULTS

(Later)

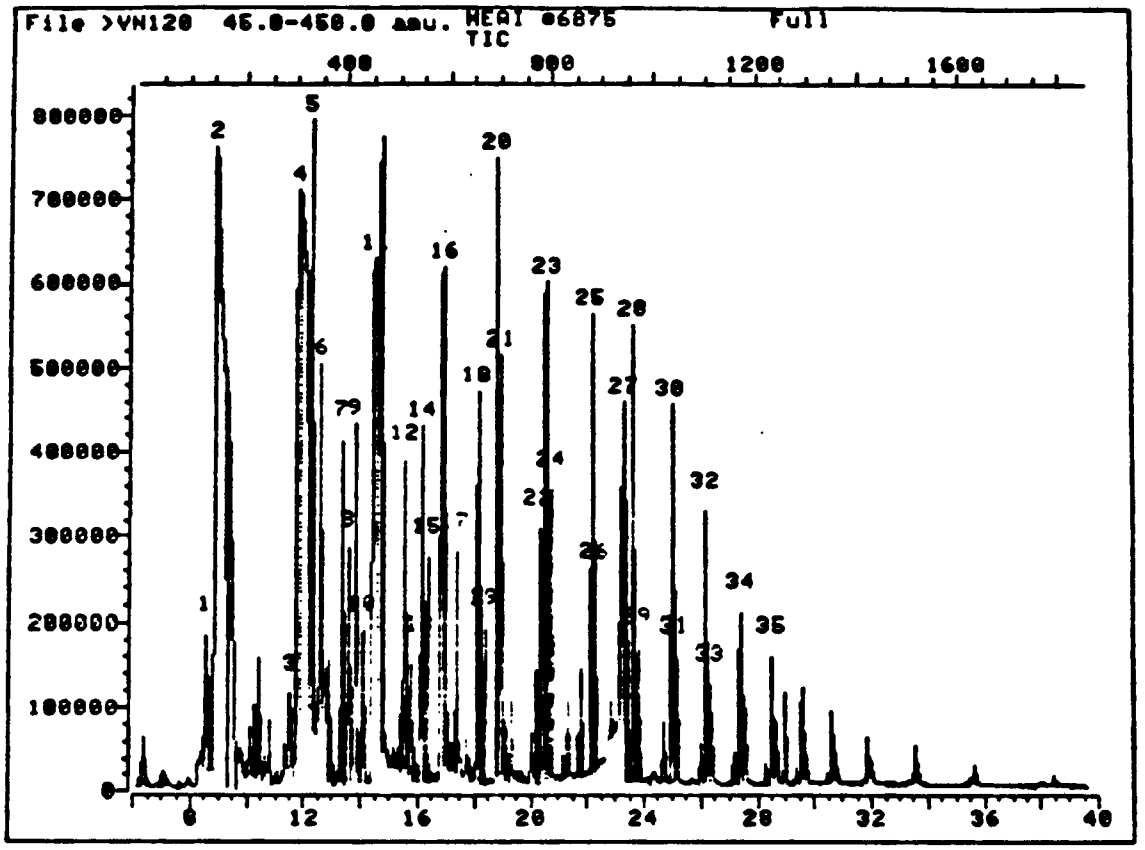


data file header from : >VN119

ple: HEAI #6866 Operator: CHUCK MS 11/19/86 2:42
c : Full BTL#12
. #: 2 MS model: 96 SW/HW rev.: IA ALS # : 0
ethod file: HE9503 Tuning file: MT9501 No. of extra records: 1
urce temp.: 190 Analyzer temp.: 200 Transfer line temp. : 250

Chromatographic temperatures :	45.	300.	0.	0.	0.
Chromatographic times, min. :	4.0	10.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	10.0	0.0	0.0	0.0	0.0

Figure 5.9-1
RECONSTRUCTED ION CHROMATOGRAPH FOR PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES OF THE OPTIMIZATION RUN NO. 1



lata file header from : >VN120

ile: HEAI #6875 Operator: CHUCK MS 11/19/86 3:32
: Full BTL#13
#: 2 MS model: 96 SW/HW rev.: IA ALS # : 0
thod file: HE9503 Tuning file: MT9501 No. of extra records: 1
irce temp.: 190 Analyzer temp.: 200 Transfer line temp. : 250

Chromatographic temperatures :	45.	300.	0.	0.	0.
Chromatographic times, min. :	4.0	10.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	10.0	0.0	0.0	0.0	0.0

Figure 5.9-2
RECONSTRUCTED ION CHROMATOGRAPH FOR PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES OF THE OPTIMIZATION RUN NO. 2

TABLE 5.9-1

SUMMARY OF IDENTIFIED PRODUCTS OF INCOMPLETE COMBUSTION
IN OFF-GAS SAMPLES OF TWO OPTIMIZATION RUNS

Compound	Total Amount Estimated (ug)	
T Primary (°C)	900	900
T Secondary (°C)	1200	1200
O_2 (%)	5.4	7
Optimization Run No.	1	2
Octamethyl Cyclotetrasiloxane	25,000	ND
1,2-Dichlorobenzene	1700	ND
Tetramethyl Pentane	540	ND
1,2,3-Trichlorobenzene	1900	ND
Chloro-4-(methylthio)-Benzene	12,000	ND
Dodicamethyl Cyclohexasiloxane	8800	8700
Tetrachlorobenzene	3000	ND
Pentachlorobenzene	3700	ND
12-Methyl-1-Tetradecanol	4000	1000
4-Methoxy-Benzoic acid Trimethylsilyl ester	670	760
Tetrachloro-5-dichloromethylene-Cyclopentadiene	770	ND
Isocyano-Naphthalene	640	ND
Pentadecyl-1, 3-Dioxolane	710	640
Ethyl-Indolecarboxylic acid ethyl ester	ND	6200
Ethyl-methyl-Pyridinethione	ND	2200
Benzeneacetic acid	ND	2200
Sulfur, mol (S8)	1600	5000

TABLE 5.10-1

EP TOXICITY TEST RESULTS

	EP Limit Concentration (mg/L)	Feed Samples (mg/l)	Residue Sample (mg/l)
As	5.0	0	0.081
Ba	100.0	0.212	0.200
Cd	1.0	0	0
Cr	5.0	0.017	0.037
Pb	5.0	0.098	0.033
Hg	0.2	0.020	0.020
Ag	5.0	0.043	0.040
Se	1.0	0.273	0.268

6.0 SUMMARY AND CONCLUSIONS

The data generated in the laboratory tests thus far conducted indicate that the toxic organics identified in the Basin F soil samples are amenable to incineration. The data suggests that a DRE of >99.99 percent can be achieved at relatively low reactor temperatures (650°C), a total gas phase residence time of approximately 7.0 seconds, and flue gas oxygen concentrations of 5-7 percent. Examination of the available literature and consideration of chemical kinetic principals suggest that most of the identified toxic organics are thermally fragile and easily decomposed.

The sulfone and sulfoxide compounds ($CPMSO_2$, CPMSO, and CPMS) appear, from a theoretical standpoint, to be the most stable toxic compounds in the waste. Oxathiane and Dithiane, which are basically hydrocarbons with thioether and ether linkages, are also expected to be moderately stable. However, it is felt that none of these materials represent a special challenge to available incineration technologies.

Analysis for products of incomplete combustion indicated that a number of chlorinated and nonchlorinated products were formed during the low temperature test runs. At higher temperatures, these compounds appeared to be destroyed and only a few products were observed. The majority of these high temperature PICs were siloxanes and partially oxidized hydrocarbons (alcohols and esters). Siloxanes may result from the thermal degradation of the stationary phase GC columns or sealing materials containing silicone rubber (such as GC septa). In light of this, one must consider the possibility that their observation is due to experimental artifact; although, their precursor may also be in the original Basin F sample. The alcohols and esters may have been formed in "cool" regions (300-500°C) of the transfer lines of the laboratory system.

The observation of chlorinated olefins and aromatics in addition to benzene, toluene, and naphthalene is as expected. These compounds have been previously shown, both experimentally and theoretically, to be thermally stable. It is surprising that such compounds as benzene, toluene,

naphthalene, and hexachlorobenzene were not observed in the high temperature runs. They were expected due to the extreme stability of hexachlorobenzene and the expected yields of the other three species. It is also surprising that some chlorinated and nonchlorinated PNAs were not observed, as previous laboratory studies have shown that they can be major products at higher temperatures.

One reason for the lack of observation of these compounds may have been that most of the off-gas GC-MS analyses were run in the selected ion monitoring (SIM) mode. This means that only a limited number of species (similar in structure to the POHCs analyzed for in the waste feed) would be observed. Still, these higher molecular weight species were not observed in the full scan PIC runs. The high molecular weight materials in question (naphthalene and hexachlorobenzene) are difficult to transport, requiring a temperature of 200-250°C to maintain them in the gas phase. If any cold spot exists in the transfer lines these species may be condensed out and not be observed in the off-gas analysis.

Consequently, it is felt that the PIC issue for Basin F wastes deserves further study. Special attention should be paid to being sure that quantitative transport of combustion products is assured. Gas chromatographic analysis using a flame ionization detector (FID) should be employed because this technique responds to a broad spectrum of organics, more completely identifying the full range of possible products. Mass spectral analysis may then be employed to analyze for specific compounds observed in the GC/FID trace as well as other suspected products of special interest such as chlorinated aromatics, PNAs, benzene, toluene, and naphthalene.

In summary, however, it is concluded that Basin F wastes are incinerable and that combustion of these wastes at the following conditions would result in the most complete oxidation.

Primary Kiln Temperature	900°C
Afterburner Temperature	1200°C
Gas Residence Time in Afterburner	2 seconds
Oxygen Level in Off-Gases	7 percent

APPENDIX A

ANALYTICAL RESULTS OF FEED, RESIDUE AND OFF-GAS SAMPLES

FEED ANALYSES

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4338 RUN#2	HEAI# 4339 RUN#3	HEAI# 4340 RUN#4	HEAI# 4341 RUN#5
		ug/g		
1) Oxathiane	<0.9	<0.9	<0.9	<0.9
2) DCPD	160	170	130	120
3) DIMP	<0.8	<0.8	<0.8	<0.8
4) DMMP	<0.8	<0.8	<0.8	<0.8
5) Dithiane	<0.3	<0.3	<0.3	<0.3
6) DBCP	41	47	35	33
7) Vapona	<0.6	<0.6	<0.6	<0.6
8) CPMS	2000	1900	1800	1500
9) HCCPD	<0.3	<0.3	<0.3	<0.3
10) CPMSO	120	140	110	95
11) CPMSO2	490	460	460	360
12) Atrazine	<3.0	<3.0	<3.0	<3.0
13) Malathion	<0.4	<0.4	<0.4	<0.4
14) Aldrin	2200	2100	2100	1700
15) Parathion	<0.6	<0.6	<0.6	<0.6
16) Isodrin	240	300	250	200
17) Supona	13	14	15	13
18) DDE	<0.9	<0.9	<0.9	<0.9
19) Dieldrin	1800	1600	1500	1200
20) Endrin	310	350	270	240
21) DDT	<0.3	<0.3	<0.3	<0.3
22) Chlordane	<2.0	<2.0	<2.0	<2.0
SURROGATE Recoveries	%	%	%	%
31) 1,3-Dichlorobenzene-d4	90	76	75	72
32) Diethylphthalate-d4	114	96	119	102
33) Dioctylphthalate-d4	126	81	100	78
34) Chlorophenol-d4	86	72	67	63

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4342 run 6	HEAI# 4343 run 7	HEAI# 4344 run 8	HEAI# 4345 run 9
		ug/g		
1) Oxathiane	<0.9	<0.9	<0.9	<0.9
2) DCPD	150	110	110	110
3) DIMP	<0.8	<0.8	<0.8	<0.8
4) DMMP	<0.8	<0.8	<0.8	<0.8
5) Dithiane	<0.3	<0.3	<0.3	<0.3
6) DBCP	36	31	28	29
7) Vapona	<0.6	<0.6	<0.6	<0.6
8) CPMS	2300	2100	2100	2100
9) HCCPD	<0.3	<0.3	<0.3	<0.3
10) CPMSO	51	57	47	47
11) CPMSO2	200	200	170	160
12) Atrazine	<3.0	<3.0	<3.0	<3.0
13) Malathion	<0.4	<0.4	<0.4	<0.4
14) Aldrin	2400	2300	1900	2300
15) Parathion	<0.6	<0.6	<0.6	<0.6
16) Isodrin	110	130	100	130
17) Supona	19	19	15	22
18) DDE	<0.9	<0.9	<0.9	<0.9
19) Dieldrin	1600	1800	1400	1800
20) Endrin	310	330	260	340
21) DDT	<0.3	<0.3	<0.3	<0.3
22) Chlordane	<2.0	<2.0	<2.0	<2.0
SURROGATE Recoveries	%	%	%	%
51) 1,3-Dichlorobenzene-d4	140	121	125	102
52) Diethylphthalate-d4	68	74	70	64
53) Dioctylphthalate-d4	136	158	130	158
54) Chlorophenol-d4	124	106	111	97

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4346 run#10	HEAI# 4347 run#11	HEAI# 4348 run#12
		ug/g	
) Oxathiane	<0.9	<0.9	<0.9
) DCPD	59	100	93
) DIMP	<0.8	<0.8	<0.8
) DMMP	<0.8	<0.8	<0.8
) Dithiane	<0.3	<0.3	<0.3
) DBCP	13	23	22
) Vapona	<0.6	<0.6	<0.6
) CPMS	1400	2100	2000
) HCCPD	<0.3	<0.3	<0.3
) CPMSO	31	53	53
) CPMSO2	150	270	250
) Atrazine	<3.0	<3.0	<3.0
) Malathion	<0.4	<0.4	<0.4
) Aldrin	1700	3600	3700
) Parathion	<0.6	<0.6	<0.6
) Isodrin	89	190	180
) Supona	7.8	17	16
) DDE	<0.9	<0.9	<0.9
) Dieldrin	1000	1800	1800
) Endrin	170	300	320
) DDT	<0.3	<0.3	<0.3
) Chlordane	<2.0	<2.0	<2.0
RROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	92	88	84
) Diethylphthalate-d4	64	62	68
) Dioctylphthalate-d4	95	85	101
) Chlorophenol-d4	102	105	100

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4349 run#13	HEAI# 4350 run#14	HEAI# 4351 run#15
	ug/g		
) Oxathiane	<0.9	<0.9	<0.9
) DCPD	70	88	95
) DIMP	<0.8	<0.8	<0.8
) DMMP	<0.8	<0.8	<0.8
) Dithiane	<0.3	<0.3	<0.3
) DBCP	15	20	24
) Vapona	<0.6	<0.6	<0.6
) CPMS	1600	1700	2000
) HCCPD	<0.3	<0.3	<0.3
) CPMSO	46	84	87
) CPMSO2	190	310	300
) Atrazine	<3.0	<3.0	<3.0
) Malathion	<0.4	<0.4	<0.4
) Aldrin	2300	3600	3300
) Parathion	<0.6	<0.6	<0.6
) Isodrin	110	220	200
) Supona	11	20	18
) DDE	<0.9	<0.9	<0.9
) Dieldrin	1100	1600	1700
) Endrin	200	390	390
) DDT	<0.3	<0.3	<0.3
) Chlordane	<2.0	<2.0	<2.0
RROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	107	68	90
) Diethylphthalate-d4	107	101	110
) Dioctylphthalate-d4	94	88	135
) Chlorophenol-d4	95	73	78

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4352 run#16	HEAI# 4353 run#17	HEAI# 4354 run#18
		ug/g	
) Oxathiane	<0.9	<0.9	<0.9
) DCPD	100	85	140
) DIMP	<0.8	<0.8	<0.8
) DMMP	<0.8	<0.8	<0.8
) Dithiane	<0.3	<0.3	<0.3
) DBCP	17	12	42
) Vapona	<0.6	<0.6	<0.6
) CPMS	2200	2100	2600
) HCCPD	<0.3	<0.3	<0.3
) CPMSO	100	100	99
) CPMSO2	350	330	330
) Atrazine	<3.0	<3.0	<3.0
) Malathion	<0.4	<0.4	<0.4
) Aldrin	3300	3100	3500
) Parathion	<0.6	<0.6	<0.6
) Isodrin	180	180	190
) Supona	17	21	22
) DDE	<0.9	<0.9	<0.9
) Dieldrin	1500	1500	1600
) Endrin	390	370	400
) DDT	<0.3	<0.3	<0.3
) Chlordane	<2.0	<2.0	<2.0
RROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	33	24	15
) Diethylphthalate-d4	81	87	79
) Dioctylphthalate-d4	145	154	142
) Chlorophenol-d4	-	-	-

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4355 run#19 ug/g	HEAI# 4356 run#20 ug/g
) Oxathiane	<0.9	<0.9
) DCPD	160	240
) DIMP	<0.8	<0.8
) DMMP	<0.8	<0.8
) Dithiane	<0.3	<0.3
) DBCP	48	49
) Vapona	<0.6	<0.6
) CPMS	2700	2600
) HCCPD	<0.3	<0.3
) CPMSO	110	91
) CPMSO2	360	280
) Atrazine	<3.0	<3.0
) Malathion	<0.4	<0.4
) Aldrin	3900	3700
) Parathion	<0.6	<0.6
) Isodrin	210	180
) Supona	26	34
) DDE	<0.9	<0.9
) Dieldrin	1800	2800
) Endrin	500	610
) DDT	<0.3	<0.3
) Chlordane	<2.0	<2.0
RROGATE Recoveries	%	%
) 1,3-Dichlorobenzene-d4	28	29
) Diethylphthalate-d4	76	74
) Dioctylphthalate-d4	152	270
) Chlorophenol-d4	-	-

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Results of Feed Soil Analysis

COMPOUND	HEAI# 4338 RUN# 2	HEAI# 4339 RUN#3	HEAI# 4340 RUN#4	HEAI# 4341 RUN#5
		Total ug		
1) Oxathiane	<320	<320	<320	<320
2) DCPD	56000	60000	46000	42000
3) DIMP	<280	<280	<280	<280
4) DMMP	<280	<280	<280	<280
5) Dithiane	<100	<100	<100	<100
6) DBCP	14000	16000	12000	12000
7) Vapona	<210	<210	<210	<210
8) CPMS	700000	660000	630000	520000
9) HCCPD	<100	<100	<100	<100
10) CPMSO	42000	49000	38000	33000
11) CPMSO2	170000	160000	160000	130000
12) Atrazine	<1000	<1000	<1000	<1000
13) Malathion	<140	<140	<140	<140
14) Aldrin	770000	740000	740000	600000
15) Parathion	<210	<210	<210	<210
16) Isodrin	84000	100000	88000	70000
17) Supona	4600	4900	5200	4600
18) DDE	<320	<320	<320	<320
19) Dieldrin	530000	560000	520000	420000
20) Endrin	100000	120000	94000	84000
21) DDT	<100	<100	<100	<100
22) Chlordane	<700	<700	<700	<700

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4342 run 6	HEAI# 4343 run 7	HEAI# 4344 run 8	HEAI# 4345 run 9
		Total ug		
1) Oxathiane	<320	<320	<320	<320
2) DCPD	52000	38000	38000	38000
3) DIMP	<290	<290	<290	<290
4) DMMP	<290	<290	<290	<290
5) Dithiane	<110	<110	<110	<110
6) DBCP	13000	11000	9800	10000
7) Vapona	<220	<220	<220	<220
8) CPMS	800000	740000	740000	740000
9) HCCPD	<180	<180	<180	<180
10) CPMSO	18000	20000	16000	16000
11) CPMSO2	70000	70000	60000	56000
12) Atrazine	<1100	<1100	<1100	<1100
13) Malathion	<140	<140	<140	<140
14) Aldrin	840000	800000	660000	800000
15) Parathion	<220	<220	<220	<220
16) Isodrin	38000	46000	35000	46000
17) Supona	6600	6600	5200	7700
18) DDE	<320	<320	<320	<320
19) Dieldrin	560000	630000	490000	630000
20) Endrin	110000	120000	91000	120000
21) DDT	<110	<110	<110	<110
22) Chlordane	<720	<720	<720	<720

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4346 run#10	HEAI# 4347 run#11	HEAI# 4348 run#12
	Total ug		
1) Oxathiane	<320	<320	<320
2) DCPD	21000	35000	33000
3) DIMP	<290	<290	<290
4) DMMP	<290	<290	<290
5) Dithiane	<110	<110	<110
6) DBCP	4600	8000	7700
7) Vapona	<220	<220	<220
8) CPMS	490000	740000	700000
9) HCCPD	<180	<180	<180
10) CPMSO	11000	19000	19000
11) CPMSO2	52000	94000	88000
12) Atrazine	<1100	<1100	<1100
13) Malathion	<140	<140	<140
14) Aldrin	600000	1300000	1300000
15) Parathion	<220	<220	<220
16) Isodrin	31000	66000	63000
17) Supona	2700	6000	5600
18) DDE	<320	<320	<320
19) Dieldrin	350000	630000	630000
20) Endrin	60000	100000	110000
21) DDT	<110	<110	<110
22) Chlordane	<720	<720	<720

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4349 run#13	HEAI# 4350 run#14	HEAI# 4351 run#15
	Total ug		
) Oxathiane	<320	<320	<320
) DCPD	24000	31000	33000
) DIMP	<290	<290	<290
) DMMP	<290	<290	<290
) Dithiane	<110	<110	<110
) DBCP	5200	7000	8400
) Vapona	<220	<220	<220
) CPMS	560000	600000	700000
) HCCPD	<180	<180	<180
) CPMSO	16000	29000	30000
) CPMSO2	66000	110000	100000
Atrazine	<1100	<1100	<1100
Malathion	<140	<140	<140
Aldrin	800000	1300000	1200000
Parathion	<220	<220	<220
Isodrin	38000	77000	70000
Supona	3800	7000	6300
DDE	<320	<320	<320
Dieldrin	380000	560000	600000
Endrin	70000	14000	14000
DDT	<110	<110	<110
Chlordane	<720	<720	<720

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4352 run#16	HEAI# 4353 run#17	HEAI# 4354 run#18
	Total ug		
) Oxathiane	<320	<320	<320
) DCPD	35000	30000	49000
) DIMP	<290	<290	<290
) DMMP	<290	<290	<290
) Dithiane	<110	<110	<110
) DBCP	6000	4200	15000
) Vapona	<220	<220	<220
) CPMS	770000	740000	910000
) HCCPD	<180	<180	<180
) CPMSO	35000	35000	35000
) CPMSO2	120000	120000	120000
) Atrazine	<1100	<1100	<1100
) Malathion	<140	<140	<140
) Aldrin	1200000	1100000	1200000
) Parathion	<220	<220	<220
) Isodrin	63000	63000	66000
) Supona	6000	7400	7700
) DDE	<320	<320	<320
) Dieldrin	520000	520000	560000
) Endrin	140000	130000	140000
) DDT	<110	<110	<110
) Chlordane	<720	<720	<720

HITTMAN EBASCO Associates

Results of Feed Soil Analysis

COMPOUND	HEAI# 4355 run#19 Total ug	HEAI# 4356 run#20 Total ug
) Oxathiane	<320	<320
) DCPD	56000	84000
) DIMP	<290	<290
) DMMP	<290	<290
) Dithiane	<110	<110
) DBCP	17000	17000
) Vapona	<220	<220
) CPMS	940000	910000
) HCCPD	<180	<180
) CPMSO	38000	32000
) CPMSO2	130000	98000
) Atrazine	<1100	<1100
) Malathion	<140	<140
) Aldrin	1400000	1300000
) Parathion	<220	<220
Isodrin	74000	63000
Supona	9100	12000
DDE	<320	<320
Dieldrin	630000	980000
Endrin	180000	210000
DDT	<110	<110
Chlordane	<720	<720

RESIDUE ANALYSES

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4595 run 2	HEAI# 4602 run 3	HEAI# 4633 run 4	HEAI# 4647 run 5
	ug/g			
1) Oxathiane	<0.008	<0.008	<0.005	<0.005
2) DCPD	<0.008	<0.008	<0.005	<0.005
3) DIMP	<0.08	<0.08	<0.05	<0.05
4) DMMP	<0.08	<0.08	<0.05	<0.05
5) Dithiane	<0.008	<0.008	<0.005	<0.005
6) DBCP	<0.008	0.020	<0.005	<0.005
7) Vapona	<0.08	<0.08	<0.05	<0.05
8) CPMS	<0.08	<0.08	<0.05	<0.05
9) HCCPD	<0.03	<0.03	<0.02	<0.02
10) CPMSO	<0.08	<0.08	2.80	<0.05
11) CPMSO2	0.25	<0.08	0.35	<0.05
12) Atrazine	<0.08	<0.08	<0.05	<0.05
13) Malathion	<0.08	<0.08	<0.05	<0.05
14) Aldrin	<0.05	<0.05	0.08	<0.03
15) Parathion	<0.08	<0.08	<0.05	<0.05
16) Isodrin	<0.08	<0.08	<0.01	<0.01
17) Supona	<0.08	<0.08	<0.05	<0.05
18) DDE	<0.05	<0.05	<0.03	<0.03
19) Dieldrin	0.15	<0.02	0.03	0.20
20) Endrin	<0.08	<0.08	<0.05	<0.05
21) DDT	<0.08	<0.08	<0.05	<0.05
22) Chlordane	<0.08	<0.08	<0.05	<0.05
SURROGATE Recoveries	%	%	%	%
51) 1,3-Dichlorobenzene-d4	61	45	46	45
52) Diethylphthalate-d4	120	112	113	115
53) Dioctylphthalate-d4	86	135	93	89
54) Chlorophenol-d4	59	44	41	40

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Results of Residue Analysis

COMPOUND	HEAI# 4656 run 6	HEAI# 4667 run 7	HEAI# 4686 run 8	HEAI# 4697 run 9
	ug/g			
1) Oxathiane	<0.005	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05	<0.05
8) CPMS	<0.05	<0.05	<0.05	<0.05
9) HCCPD	<0.02	<0.02	<0.02	<0.02
0) CPMSO	<0.05	<0.05	0.06	<0.05
1) CPMSO2	<0.05	<0.05	<0.05	<0.05
2) Atrazine	<0.05	<0.05	<0.05	<0.05
3) Malathion	<0.05	<0.05	<0.05	<0.05
4) Aldrin	<0.03	0.03	0.18	0.05
5) Parathion	<0.05	<0.05	<0.05	<0.05
6) Isodrin	<0.05	<0.05	<0.01	<0.01
7) Supona	<0.05	<0.05	<0.05	<0.05
8) DDE	<0.03	<0.03	<0.03	<0.03
9) Dieldrin	<0.01	<0.01	0.10	<0.01
0) Endrin	<0.05	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05	<0.05
URROGATE Recoveries	%	%	%	%
1) 1,3-Dichlorobenzene-d4	78	90	97	68
2) Diethylphthalate-d4	83	104	106	85
3) Dioctylphthalate-d4	119	112	145	107
4) Chlorophenol-d4	83	104	107	72

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4715 run 10	HEAI# 4725 run 11	HEAI# 4753 run 12
		ug/g	
1) Oxathiane	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05
8) CPMS	0.08	<0.05	<0.05
9) HCCPD	<0.02	<0.02	<0.02
0) CPMSO	<0.05	<0.05	<0.05
1) CPMSO2	0.05	4.0	0.87
2) Atrazine	<0.05	<0.05	<0.05
3) Malathion	<0.05	<0.05	<0.05
4) Aldrin	0.23	3.0	0.05
5) Parathion	<0.05	<0.05	<0.05
6) Isodrin	<0.01	0.04	<0.01
7) Supona	<0.05	<0.05	<0.05
8) DDE	<0.03	<0.03	<0.03
9) Dieldrin	0.10	2.8	0.03
0) Endrin	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05
IRROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	64	101	60
2) Diethylphthalate-d4	51	53	55
3) Dioctylphthalate-d4	50	99	45
4) Chlorophenol-d4	98	138	99

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Results of Residue Analysis

COMPOUND	HEAI# 4763 run 13	HEAI# 4785 run 14	HEAI# 4799 run 15
		ug/g	
1) Oxathiane	<0.005	<0.005	<0.25
2) DCPD	<0.005	<0.005	3.0
3) DIMP	<0.05	<0.05	<2.5
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.25
6) DBCP	<0.005	<0.005	0.94
7) Vapona	<0.05	<0.05	<2.5
8) CPMS	<0.05	1.1	2500
9) HCCPD	<0.02	<0.02	<1.0
0) CPMSO	<0.05	<0.05	5300
1) CPMSO2	<0.05	0.10	864
2) Atrazine	<0.05	<0.05	<2.5
3) Malathion	<0.05	<0.05	<2.5
4) Aldrin	<0.03	0.07	1700
5) Parathion	<0.05	<0.05	<2.5
6) Isodrin	<0.01	<0.01	160
7) Supona	<0.05	<0.05	14
8) DDE	<0.03	<0.03	<1.5
9) Dieldrin	<0.02	0.04	2200
0) Endrin	<0.05	<0.05	82
1) DDT	<0.05	<0.05	<2.5
2) Chlordane	<0.05	<0.05	<2.5
RROGATE Recoveries	%	%	%
1) 1,3-Dichlorobenzene-d4	51	54	92
2) Diethylphthalate-d4	96	97	43
3) Dioctylphthalate-d4	80	82	82
4) Chlorophenol-d4	79	80	103

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4961 run#16	HEAI# 5155 run#17	HEAI# 5190 run#18
		ug/g	
) Oxathiane	<0.005	<0.005	<0.005
) DCPD	<0.005	<0.005	<0.005
) DIMP	<0.05	<0.05	<0.05
) DMMP	<0.05	<0.05	<0.05
) Dithiane	<0.005	<0.005	<0.005
) DBCP	<0.005	<0.005	<0.005
) Vapona	<0.05	<0.05	<0.05
) CPMS	17	<0.05	<0.05
) HCCPD	<0.02	<0.02	<0.02
) CPMSO	<0.05	<0.05	<0.05
) CPMSO2	2.7	<0.05	<0.05
) Atrazine	<0.05	<0.05	<0.05
) Malathion	<0.05	<0.05	<0.05
) Aldrin	16	0.25	0.81
) Parathion	<0.05	<0.05	<0.05
) Isodrin	0.27	<0.01	<0.01
) Supona	0.10	<0.05	<0.05
) DDE	<0.03	<0.03	<0.03
) Dieldrin	9.2	0.06	0.93
) Endrin	0.38	<0.05	<0.05
) DDT	<0.05	<0.05	<0.05
) Chlordane	<0.05	<0.05	<0.05
RROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	83	45	40
) Diethylphthalate-d4	60	65	53
) Dioctylphthalate-d4	155	83	149
) Chlorophenol-d4	34	48	39

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 5247 run#19	HEAI# 5275 run#20	HEAI# 5297 run#21
		ug/g	
1) Oxathiane	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05
8) CPMS	<0.05	0.06	<0.05
9) HCCPD	<0.02	<0.02	<0.02
0) CPMSO	<0.05	<0.05	<0.05
1) CPMSO2	<0.05	<0.05	<0.05
2) Atrazine	<0.05	<0.05	<0.05
3) Malathion	<0.05	<0.05	<0.05
4) Aldrin	<0.03	<0.03	0.09
5) Parathion	<0.05	<0.05	<0.05
6) Isodrin	<0.01	<0.01	<0.01
7) Supona	<0.05	<0.05	<0.05
8) DDE	<0.03	<0.03	<0.03
9) Dieldrin	<0.02	<0.02	0.03
0) Endrin	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05
URROGATE Recoveries	%	%	%
1) 1,3-Dichlorobenzene-d4	60	97	85
2) Diethylphthalate-d4	62	78	70
3) Dioctylphthalate-d4	92	105	85
4) Chlorophenol-d4	66	119	88

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4595 run 2	HEAI# 4602 run 3	HEAI# 4633 run 4	HEAI# 4647 run 5
	Total ug			
1) Oxathiane	<1.5	<1.6	<0.88	<0.92
2) DCPD	<1.5	<1.6	<0.88	<0.92
3) DIMP	< 15	< 16	< 8.8	< 9.2
4) DMMP	< 15	< 16	< 8.8	< 9.2
5) Dithiane	<1.5	<1.6	<0.88	<0.92
6) DBCP	<1.5	3.9	<0.88	<0.92
7) Vapona	< 15	< 16	< 8.8	< 9.2
8) CPMS	< 15	< 16	< 8.8	< 9.2
9) HCCPD	<5.5	<5.8	< 3.5	< 3.7
10) CPMSO	< 15	< 16	490	< 9.2
11) CPMSO2	46	< 16	61	< 9.2
12) Atrazine	< 15	< 16	< 8.8	< 9.2
13) Malathion	< 15	< 16	< 8.8	< 9.2
14) Aldrin	<9.2	<9.7	14	< 5.5
15) Parathion	< 15	< 16	< 8.8	< 9.2
16) Isodrin	< 15	< 16	< 8.8	< 9.2
17) Supona	< 15	< 16	< 8.8	< 9.2
18) DDE	<9.2	<9.7	< 5.2	< 5.5
19) Dieldrin	27	<3.9	5.2	37
20) Endrin	< 15	< 16	< 8.8	< 9.2
21) DDT	< 15	< 16	< 8.8	< 9.2
22) Chlordane	< 15	< 16	< 8.8	< 9.2

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4656 run 6	HEAI# 4667 run 7	HEAI# 4686 run 8	HEAI# 4697 run 9
	Total ug			
1) Oxathiane	<0.78	<0.92	<0.90	<0.83
2) DCPD	<0.78	<0.92	<0.90	<0.83
3) DIMP	< 7.8	< 9.2	< 9.0	< 8.3
4) DMMP	< 7.8	< 9.2	< 9.0	< 8.3
5) Dithiane	<0.78	<0.92	<0.90	<0.83
6) DBCP	<0.78	<0.92	<0.90	<0.83
7) Vapona	< 7.8	< 9.2	< 9.0	< 8.3
8) CPMS	< 7.8	< 9.2	< 9.0	< 8.3
9) HCCPD	< 3.1	< 3.7	< 3.6	< 3.3
10) CPMSO	< 7.8	< 9.2	11	< 8.3
11) CPMSO2	< 7.8	< 9.2	< 9.0	< 8.3
12) Atrazine	< 7.8	< 9.2	< 9.0	< 8.3
13) Malathion	< 7.8	< 9.2	< 9.0	< 8.3
14) Aldrin	< 3.1	5.6	33	8.3
15) Parathion	< 7.8	< 9.2	< 9.0	< 8.3
16) Isodrin	< 1.6	< 1.8	< 1.8	< 1.7
17) Supona	< 7.8	< 9.2	< 9.0	< 8.3
18) DDE	< 3.1	< 3.7	< 3.6	< 3.3
19) Dieldrin	< 1.6	< 1.8	18	< 1.7
20) Endrin	< 7.8	< 9.2	< 9.0	< 8.3
21) DDT	< 7.8	< 9.2	< 9.0	< 8.3
22) Chlordane	< 7.8	< 9.2	< 9.0	< 8.3

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4715 run 10	HEAI# 4725 run 11	HEAI# 4753 run 12
	Total ug		
1) Oxathiane	<0.7	<0.7	<0.7
2) DCPD	<0.7	<0.7	<0.7
3) DIMP	<7.5	<7.5	<7.5
4) DMMP	<7.5	<7.5	<7.5
5) Dithiane	<0.7	<0.7	<0.7
6) DBCP	<0.7	<0.7	<0.7
7) Vapona	<7.5	<7.5	<7.5
8) CPMS	12	<7.5	<7.5
9) HCCPD	<2.6	<2.6	<2.6
0) CPMSO	<7.5	<7.5	<7.5
1) CPMSO2	7.5	600	130
2) Atrazine	<7.5	<7.5	<7.5
3) Malathion	<7.5	<7.5	<7.5
4) Aldrin	34	450	7.5
5) Parathion	<7.5	<7.5	<7.5
6) Isodrin	<1.5	6.0	<1.5
7) Supona	<7.5	<7.5	<7.5
8) DDE	<3.6	<3.6	<3.6
9) Dieldrin	15	420	4.5
0) Endrin	<7.5	<7.5	<7.5
1) DDT	<7.5	<7.5	<7.5
2) Chlordane	<7.5	<7.5	<7.5

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4763 run 13	HEAI# 4785 run 14	HEAI# 4799 run 15
	Total ug		
1) Oxathiane	<0.7	<0.7	<0.7
2) DCPD	<0.7	<0.7	450
3) DIMP	<7.5	<7.5	<7.5
4) DMMP	<7.5	<7.5	<7.5
5) Dithiane	<0.7	<0.7	<0.7
6) DBCP	<0.7	<0.7	140
7) Vapona	<7.5	<7.5	<7.5
8) CPMS	<7.5	160	380000
9) HCCPD	<2.6	<2.6	<2.6
0) CPMSO	<7.5	<7.5	800000
1) CPMSO2	<7.5	15	130000
2) Atrazine	<7.5	<7.5	<7.5
3) Malathion	<7.5	<7.5	<7.5
4) Aldrin	<3.6	10	260000
5) Parathion	<7.5	<7.5	<7.5
6) Isodrin	<7.5	<7.5	24000
7) Supona	<7.5	<7.5	2100
8) DDE	<3.6	<3.6	<3.6
9) Dieldrin	<1.8	6.0	330000
0) Endrin	<7.5	<7.5	12000
1) DDT	<7.5	<7.5	<7.5
2) Chlordane	<7.5	<7.5	<7.5

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 4961 run#16 190 gm	HEAI# 5155 run#17 184 gm	HEAI# 5190 run#18 183 gm
	Total ug		
) Oxathiane	<0.95	<0.92	<0.92
) DCPD	<0.95	<0.92	<0.92
) DIMP	<9.5	<9.2	<9.2
) DMMP	<9.5	<9.2	<9.2
) Dithiane	<0.95	<0.92	<0.92
) DBCP	<0.95	<0.92	<0.92
) Vapona	<9.5	<9.2	<9.2
) CPMS	3230	<9.2	<9.2
) HCCPD	<3.8	<3.8	<3.8
) CPMSO	<9.5	<9.2	<9.2
) CPMSO2	510	<9.2	<9.2
Atrazine	<9.5	<9.2	<9.2
Malathion	<9.5	<9.2	<9.2
Aldrin	3000	46	150
Parathion	<9.5	<9.2	<9.2
Isodrin	51	<1.8	<1.8
Supona	19	<9.2	<9.2
DDE	<5.7	<5.5	<5.5
Dieldrin	1700	11	1.7
Endrin	72	<9.2	<9.2
DDT	<9.5	<9.2	<9.2
Chlordane	<9.5	<9.2	<9.2

HITTMAN EBASCO Associates

Results of Residue Analysis

COMPOUND	HEAI# 5247 run#19 187 gm	HEAI# 5275 run#20 186 gm	HEAI# 5297 run#21 136 gm
	Total ug		
) Oxathiane	<0.93	<0.93	<0.68
) DCPD	<0.93	<0.93	<0.68
) DIMP	<9.3	<9.3	<6.8
) DMMP	<9.3	<9.3	<6.8
) Dithiane	<0.93	<0.93	<0.68
) DBCP	<0.93	<0.93	<0.68
) Vapona	<9.3	<9.3	<6.8
) CPMS	<9.3	11	<6.8
) HCCPD	<3.7	<3.7	<2.7
) CPMSO	<9.3	<9.3	<6.8
) CPMSO2	<9.3	<9.3	<6.8
) Atrazine	<9.3	<9.3	<6.8
) Malathion	<9.3	<9.3	<6.8
) Aldrin	<5.6	<5.6	12
) Parathion	<9.3	<9.3	<6.8
) Isodrin	<1.9	<1.9	<1.4
) Supona	<9.3	<9.3	<6.8
) DDE	<5.6	<5.6	<4.1
) Dieldrin	<3.7	<3.7	4.1
) Endrin	<9.3	<9.3	<6.8
) DDT	<9.3	<9.3	<6.8
) Chlordane	<9.3	<9.3	<6.8

OFF-GASES ANALYSES

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 4599 run 2	HEAI# 4603 run 3	HEAI# 4638 run 4	HEAI# 4651 run 5
	Total ug			
1) Oxathiane	<0.005	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005	<0.005
6) DBCP	1.1	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05	<0.05
8) CPMS	27	57	<0.05	<0.05
9) HCCPD	<0.02	<0.02	<0.02	<0.02
.0) CPMSO	<0.05	290	<0.05	<0.05
.1) CPMSO2	3.2	<0.05	.35	<0.05
.2) Atrazine	<0.05	<0.05	<0.05	<0.05
.3) Malathion	<0.05	<0.05	<0.05	<0.05
4) Aldrin	21	480	<0.03	<0.03
5) Parathion	<0.05	<0.05	<0.05	<0.05
6) Isodrin	0.50	8.8	<0.01	<0.01
7) Supona	<0.05	<0.05	<0.05	<0.05
8) DDE	<0.03	<0.03	<0.03	<0.03
9) Dieldrin	23	190	.26	<0.01
0) Endrin	<0.05	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05	<0.05
URROGATE Recoveries	%	%	%	%
1) 1,3-Dichlorobenzene-d4	79	54	67	98
2) Diethylphthalate-d4	89	65	81	117
3) Dioctylphthalate-d4	92	106	76	88
4) Chlorophenol-d4	75	56	49	112

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 4658 run 6	HEAI# 4669 run 7	HEAI# 4688 run 8	HEAI# 4699 run 9
		Total ug		
1) Oxathiane	<0.005	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05	<0.05
8) CPMS	<0.05	0.13	<0.05	6.44
9) HCCPD	<0.02	<0.02	<0.02	<0.02
0) CPMSO	<0.05	<0.05	<0.05	<0.05
1) CPMSO2	<0.05	<0.05	<0.05	1.94
2) Atrazine	<0.05	<0.05	<0.05	<0.05
3) Malathion	<0.05	<0.05	<0.05	<0.05
4) Aldrin	<0.03	0.14	<0.03	<0.03
5) Parathion	<0.05	<0.05	<0.05	<0.05
6) Isodrin	<0.01	0.10	<0.01	0.85
7) Supona	<0.05	<0.05	<0.05	<0.05
8) DDE	<0.03	<0.03	<0.03	<0.03
9) Dieldrin	<0.01	0.11	<0.01	25
0) Endrin	<0.05	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05	<0.05
URROGATE Recoveries	%	%	%	%
1) 1,3-Dichlorobenzene-d4	98	43	39	40
2) Diethylphthalate-d4	54	105	58	53
3) Dioctylphthalate-d4	48	92	82	108
4) Chlorophenol-d4	33	44	44	102

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 4717 run 10	HEAI# 4727 run 11	HEAI# 4755 run 12
		Total ug	
1) Oxathiane	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	<0.005
7) Vapona	<0.05	<0.05	<0.05
8) CPMS	<0.05	<0.05	<0.05
9) HCCPD	<0.02	<0.02	<0.02
0) CPMSO	<0.05	<0.05	<0.05
1) CPMSO2	<0.05	<0.05	<0.05
2) Atrazine	<0.05	<0.05	<0.05
3) Malathion	<0.05	<0.05	<0.05
4) Aldrin	<0.03	<0.03	<0.03
5) Parathion	<0.05	<0.05	<0.05
6) Isodrin	<0.01	<0.01	<0.01
7) Supona	<0.05	37	<0.05
8) DDE	<0.03	<0.03	<0.03
9) Dieldrin	0.36	3.5	<0.01
0) Endrin	<0.05	<0.05	<0.05
1) DDT	<0.05	<0.05	<0.05
2) Chlordane	<0.05	<0.05	<0.05
URROGATE Recoveries	%	%	%
.) 1,3-Dichlorobenzene-d4	130	107	123
2) Diethylphthalate-d4	72	52	58
3) Dioctylphthalate-d4	80	101	64
4) Chlorophenol-d4	106	4	87

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 4765 run 13	HEAI# 4787 run 14	HEAI# 4801 run 15
		Total ug	
) Oxathiane	<0.005	<0.005	<0.005
) DCPD	<0.005	2.9	<0.005
) DIMP	<0.05	<0.05	<0.05
) DMMP	<0.05	<0.05	<0.05
) Dithiane	<0.005	<0.005	<0.005
) DBCP	<0.005	<0.005	<0.005
) Vapona	<0.05	<0.05	<0.05
) CPMS	<0.05	<0.05	<0.05
) HCCPD	<0.02	<0.02	<0.02
) CPMSO	<0.05	<0.05	<0.05
) CPMSO2	<0.05	1.4	<0.05
) Atrazine	<0.05	<0.05	<0.05
) Malathion	<0.05	<0.05	<0.05
) Aldrin	<0.03	0.7	1.7
) Parathion	<0.05	<0.05	<0.05
) Isodrin	<0.01	<0.01	<0.01
) Supona	<0.05	18	<0.05
) DDE	<0.03	<0.03	<0.03
) Dieldrin	<0.01	1.2	3.0
) Endrin	<0.05	<0.05	<0.05
) DDT	<0.05	<0.05	<0.05
) Chlordane	<0.05	<0.05	<0.05
RROGATE Recoveries	%	%	%
) 1,3-Dichlorobenzene-d4	59	107	132
) Diethylphthalate-d4	68	26	52
) Dioctylphthalate-d4	49	48	122
) Chlorophenol-d4	58	56	89

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 4963 run#16	HEAI# 5157 run#17	HEAI# 5192 run#18
		Total ug	
1) Oxathiane	<0.005	<0.005	<0.005
2) DCPD	3.2	<0.005	<0.005
3) DIMP	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005
6) DBCP	<0.005	<0.005	1.8
7) Vapona	<0.05	<0.05	<0.05
8) CPMS	<0.05	<0.05	3.8
9) HCCPD	<0.02	<0.02	<0.02
10) CPMSO	<0.05	<0.05	<0.05
11) CPMSO2	<0.05	<0.05	<0.05
12) Atrazine	<0.05	<0.05	<0.05
13) Malathion	<0.05	<0.05	<0.05
14) Aldrin	3.2	2.0	0.49
15) Parathion	<0.05	<0.05	<0.05
16) Isodrin	0.38	0.39	<0.01
17) Supona	0.75	<0.05	<0.05
18) DDE	<0.03	<0.03	<0.03
19) Dieldrin	<0.01	<0.01	0.25
20) Endrin	<0.05	<0.05	0.20
21) DDT	<0.05	<0.05	<0.05
22) Chlordane	<0.05	<0.05	<0.05
SURROGATE Recoveries	%	%	%
S1) 1,3-Dichlorobenzene-d4	37	37	66
S2) Diethylphthalate-d4	63	88	52
S3) Dioctylphthalate-d4	73	73	71
S4) Chlorophenol-d4	33	29	57

HITTMAN EBASCO Associates

Composite of Charcoal, XAD-2, and filter

COMPOUND	HEAI# 5249 run#19	HEAI# 5277 run#20	HEAI# 5300 run#21
		Total ug	
1) Oxathiane	<0.005	<0.005	<0.005
2) DCPD	<0.005	<0.005	490
3) DIMP	<0.05	<0.05	<0.05
4) DMMP	<0.05	<0.05	<0.05
5) Dithiane	<0.005	<0.005	<0.005
6) DBCP	0.16	<0.005	930
7) Vapona	<0.05	<0.05	<0.05
8) CPMS	<0.05	2.0	<0.05
9) HCCPD	<0.02	<0.02	<0.02
10) CPMSO	<0.05	<0.05	<0.05
11) CPMSO2	<0.05	12	<0.05
12) Atrazine	<0.05	<0.05	<0.05
13) Malathion	<0.05	<0.05	<0.05
14) Aldrin	0.30	<0.03	1500
15) Parathion	<0.05	<0.05	<0.05
16) Isodrin	<0.01	<0.01	<0.01
17) Supona	<0.05	<0.05	<0.05
18) DDE	<0.03	<0.03	<0.03
) Dieldrin	0.94	0.55	22
) Endrin	3.7	4.6	<0.05
21) DDT	<0.05	<0.05	<0.05
22) Chlordane	<0.05	<0.05	<0.05
SURROGATE Recoveries	%	%	%
S1) 1,3-Dichlorobenzene-d4	47	61	62
S2) Diethylphthalate-d4	39	60	55
S3) Dioctylphthalate-d4	71	97	127
S4) Chlorophenol-d4	63	91	73

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Blank Sample Results

COMPOUND	HEAI QC# 0828C	HEAI QC# 0902A
1) Oxathiane	<0.005	<0.005
2) DCPD	<0.005	<0.005
3) DIMP	<0.05	<0.05
4) DMMP	<0.05	<0.05
5) Dithiane	<0.005	<0.005
6) DBCP	<0.005	<0.005
7) Vapona	<0.05	<0.05
8) CPMS	<0.05	<0.05
9) HCCPD	<0.02	<0.02
10) CPMSO	<0.05	<0.05
11) CPMSO2	<0.05	<0.05
12) Atrazine	<0.05	<0.05
13) Malathion	<0.05	<0.05
14) Aldrin	<0.03	<0.03
15) Parathion	<0.05	<0.05
16) Isodrin	<0.01	<0.01
17) Supona	<0.05	<0.05
18) DDE	<0.03	<0.03
19) Dieldrin	<0.01	<0.01
20) Endrin	<0.05	<0.05
21) DDT	<0.05	<0.05
22) Chlordane	<0.05	<0.05
SURROGATE Recoveries	%	%
S1) 1,3-Dichlorobenzene-d4	70	59
S2) Diethylphthalate-d4	64	69
S3) Dioctylphthalate-d4	88	86
S4) Chlorophenol-d4	81	62

APPENDIX B

CHEMICAL STRUCTURES OF 22 SEMIVOLATILE ORGANIC TARGET COMPOUNDS

CROSS REFERENCE TO STRUCTURES OF COMPOUNDS TESTED FOR IN THE BENCH SCALE LABORATORY TEST PROGRAM

1. **Oxathiane** - Thioxane, C_4H_8SO, 1,4-Oxathiane

2. **DCPD** - dicyclopentadiene, $C_{10}H_{12}$, 3a,4,7,7a-Tetrahydro-4,7-methanoindene

3. **DIMP** - diisopropylmethylphosphonate, $C_7H_{17}O_3P$

4. **DMMP** - dimethylmethylphosphonate, $C_3H_9O_3P$

5. **Dithiane** - Nabam, $C_4H_6N_2Na_2S_4$, Ethylenebis(dithiocarbamic acid)disodium salt

6. **DBCP** - Nemagon, dibromochloropropane, $C_3H_5Br_2Cl$, 3-Chloro-1,2-dibromopropane

7. **Vapona** - dichlorvos, $C_4H_7Cl_2PO_4$ O,O-dimethyl O-(2,2-dichlorovinyl phosphate)

8. **PCPMS** - p-chlorophenylmethylsulfide, C_7H_7ClS

9. **HCPD** - C_5Cl_6, hexachlorocyclopentadiene

10. **PCPMSO** - p-chlorophenylmethylsulfoxide, C_7H_7ClSO

11. **$PCPMSO_2$** - p-chlorophenylmethylsulfone, $C_7H_7ClSO_2$

12. **Atrazine** - $C_8H_{14}N_5Cl$, 2-chloro-4-ethylamino-6-isopropylamino-s-triazine

13. **Malathion** - $C_{10}H_{19}O_6PS_2$, S - (1-2 dicarbethoxyethyl)O,O-dimethyldithiophosphate

14. **Aldrin** - $C_{12}H_8Cl_6$, 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4:5,8-dimethanonaphthalene

15. **Parathion** - $C_{10}H_{14}NO_5PS$, O,O-dietlyl O-p-nitrophenyl phosphorothioate

16. **Isodrin** - $C_{12}H_8Cl_6$, 1,2,3,4,10,10-hexachloro-1,4,4a,8,8a hexahydro-1,4:5,8-endo-dimethanonaphthalene

17. **Supona** - Chlorfenvinphos, $C_{12}H_{14}Cl_3O_3P$, O,O-diethyl O-[2-chloro-1-(2,4-dichlorophenyl)vinyl] phosphate

18. **P,P'-DDE** - $C_{14}H_8Cl_4$, 1,1-dichloro-2,2-bis-(p-chlorophenyl)ethylene

CROSS REFERENCE TO STRUCTURES OF COMPOUNDS TESTED FOR IN THE BENCH SCALE LABORATORY TEST PROGRAM (Continued)

19. Dieldrin - $C_{12}H_8Cl_6O$, 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4,5,8-dimethanonaphthalene

20. Endrin - $C_{12}H_8Cl_6O$, 1,2,3,4,10,10-hexachloro-6,7-epoxy, 1,4,4a,5,6,7,8,8a-octahydro-endo-endo-1,4:5,8-dimethanonaphthalene

21. P,P'-DDT - dichloro diphenyl trichloroethane $(C_1C_6H_4)_2CHCCl_3$, 1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane

22. Chlordane - $C_{10}H_6Cl_8$, 1,2,4,5,6,7,8,8-octachloro-4,7,methane-3a,4,7,7a-tetrahydroindane

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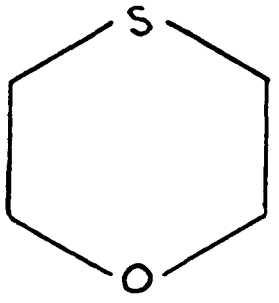
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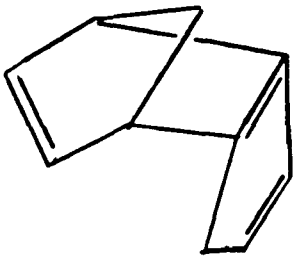

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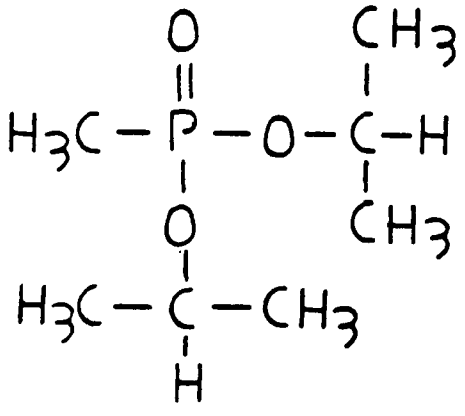
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CH3
H3C – P – O – C – H
O
CH3
H3C – C – CH3
H

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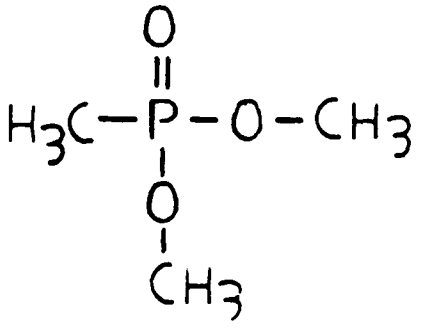
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H3C – P – O – CH3
O
CH3

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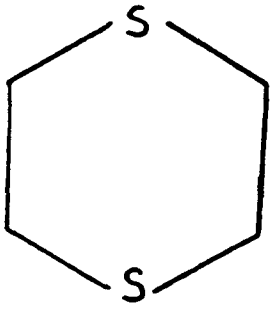
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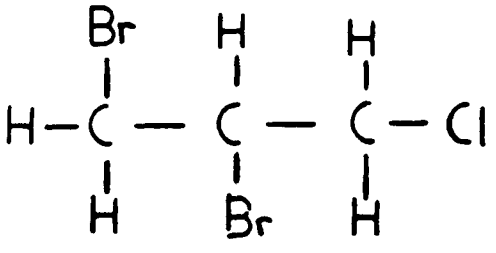
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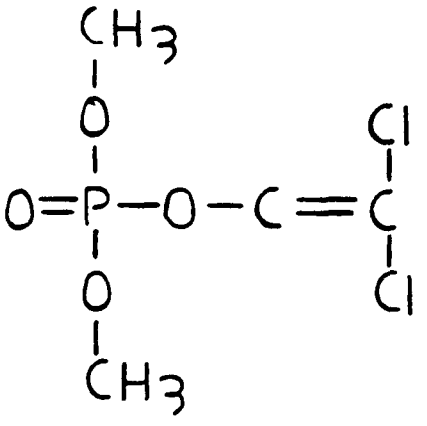
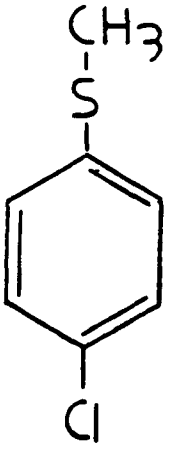
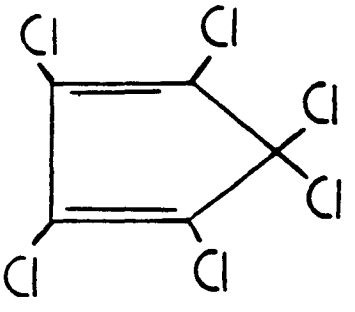
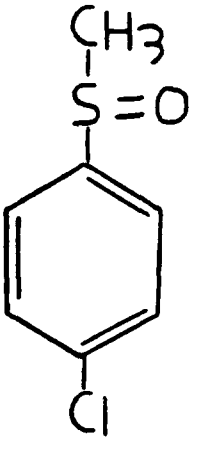
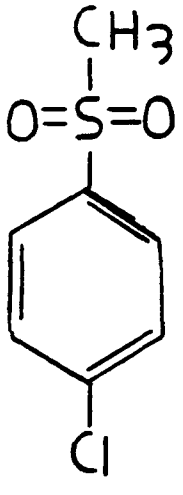
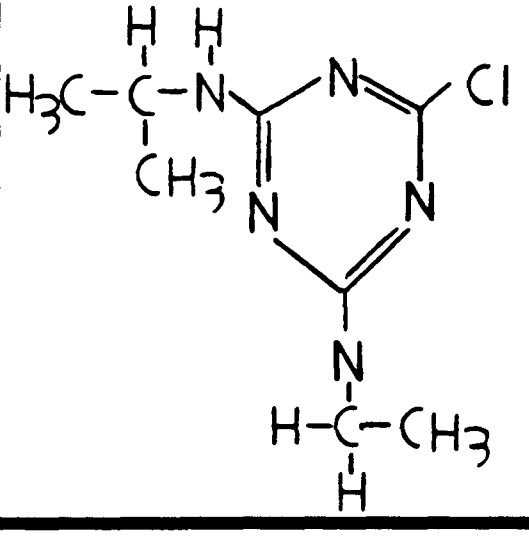
H
H
H – C – C – C – Cl
H
Br
H

7 	**8** 
9 	**10** 
11 	**12** 

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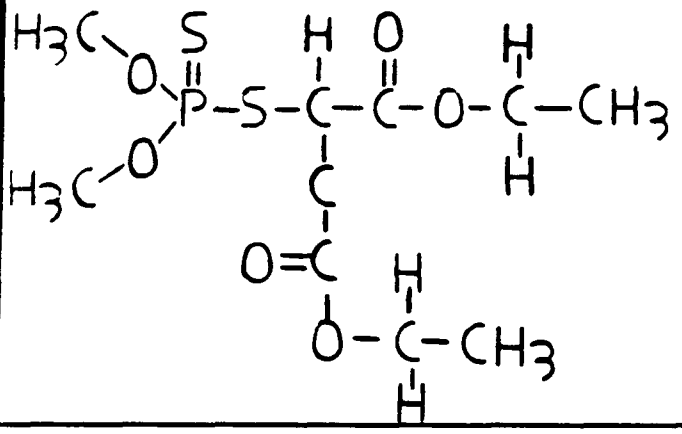

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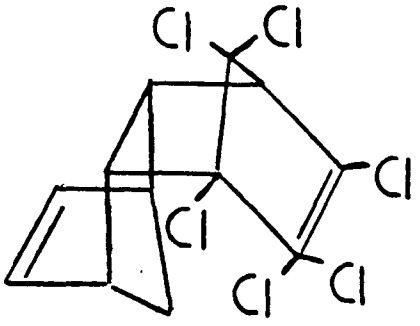

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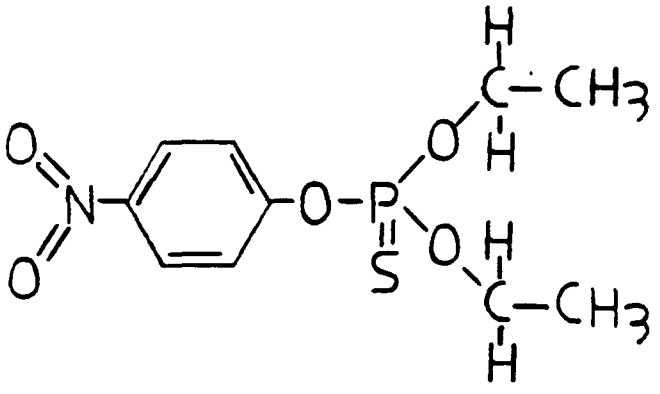

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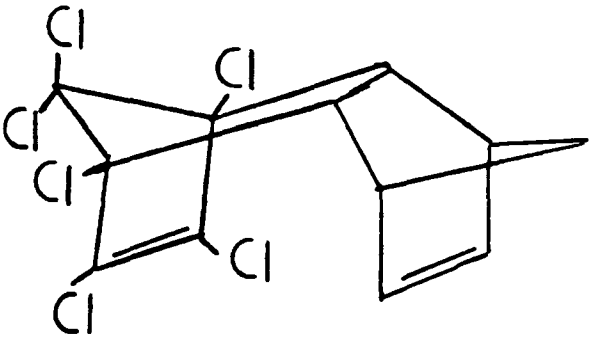

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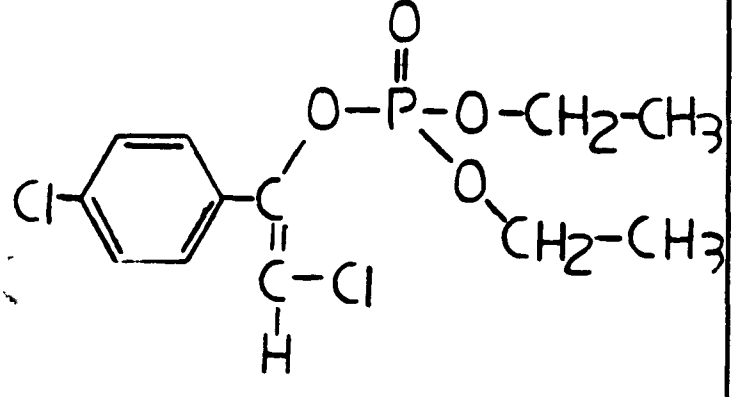

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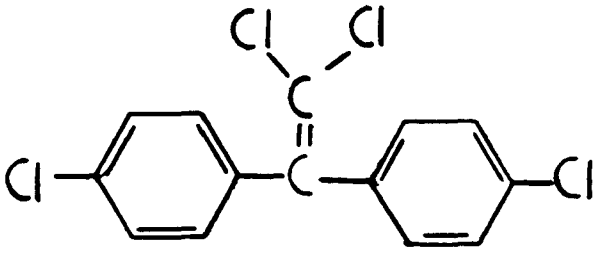

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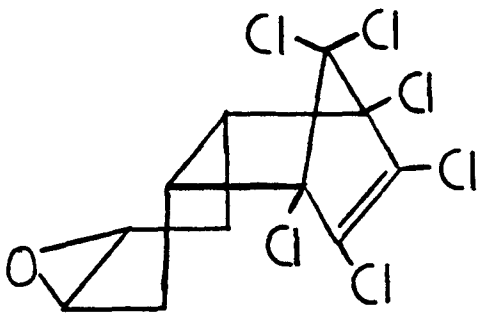

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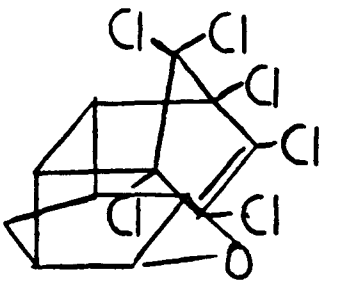

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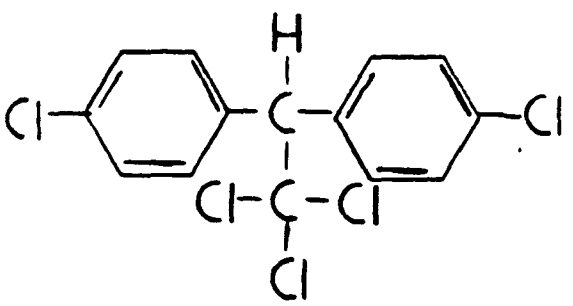

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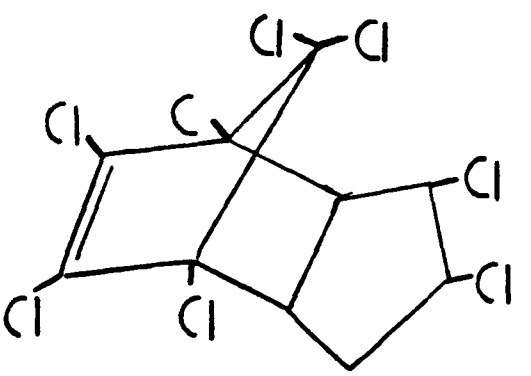

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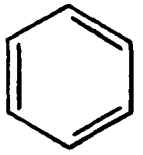
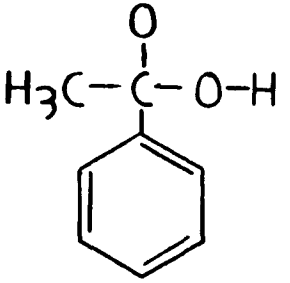
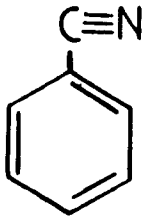
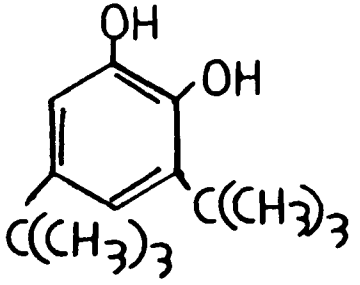
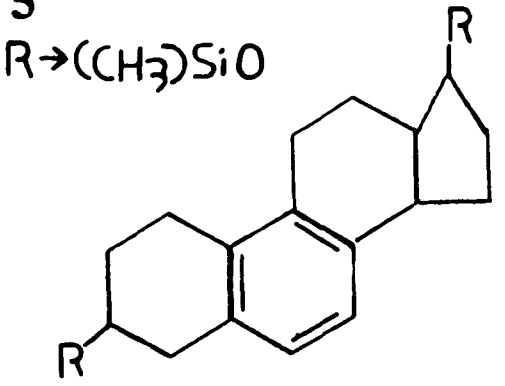
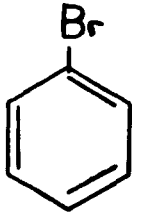
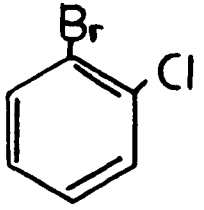
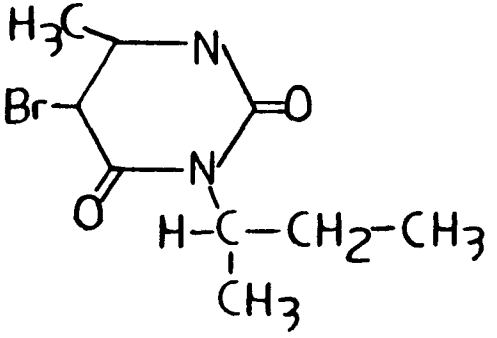
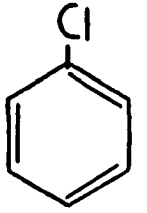
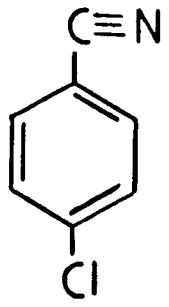
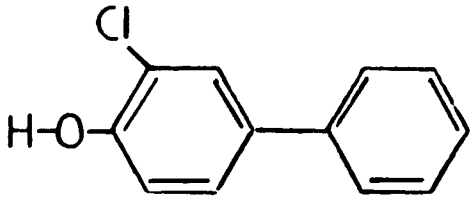
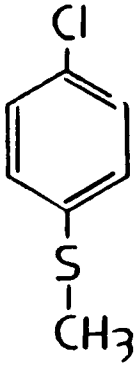
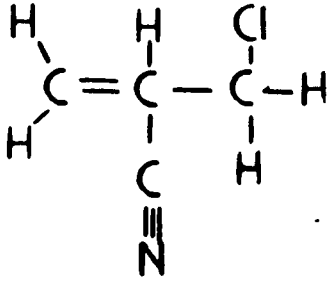
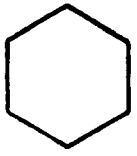
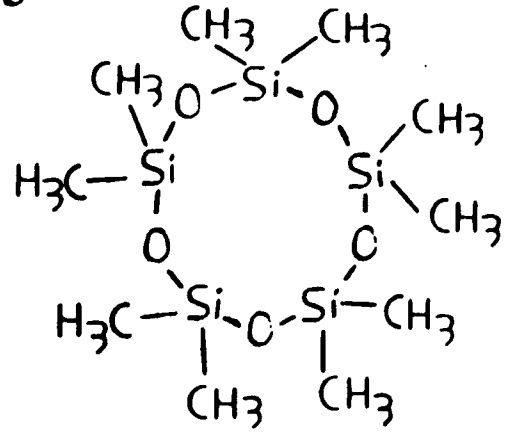
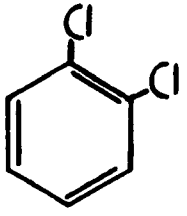
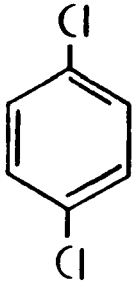
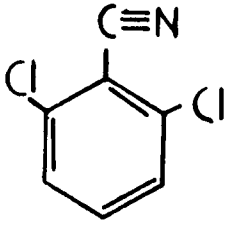
APPENDIX C

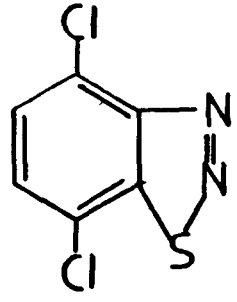
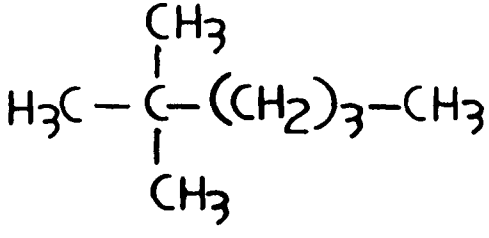
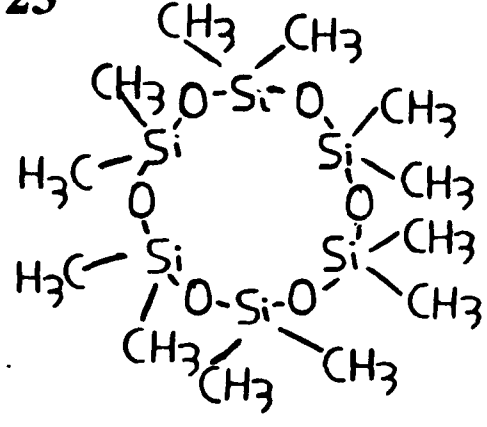
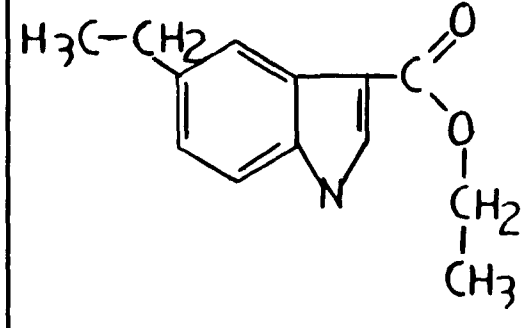
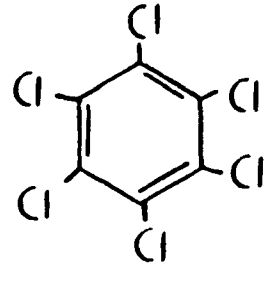
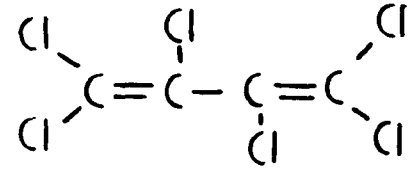
CHEMICAL STRUCTURES OF IDENTIFIED PRODUCTS OF INCOMPLETE COMBUSTION IN OFF-GASES FROM RUN NOS. 12, 13, AND 14

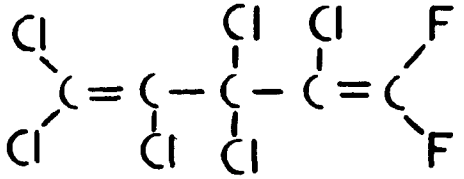
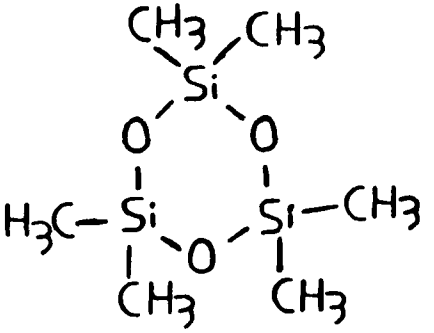
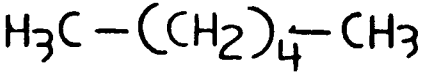
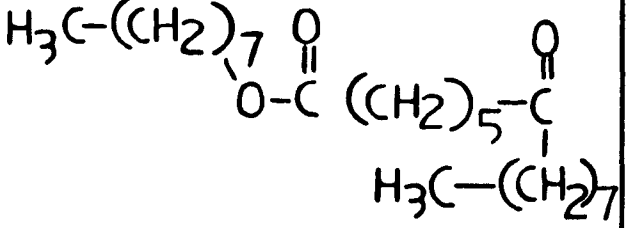
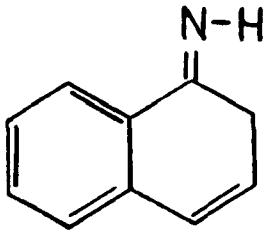
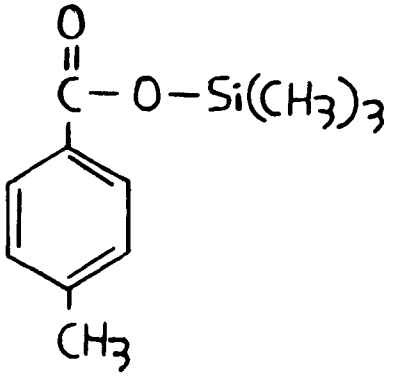
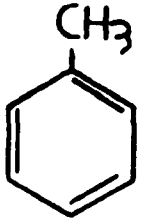
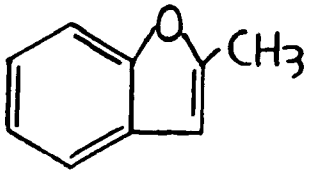
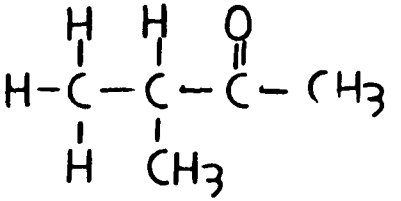
ALPHABETIZED CROSS-REFEREN[illegible]TO THE STRUCTURES
OF PRODUCTS OF INCOMPLETE [illegible]OMBUSTION (PICs)

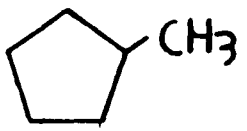
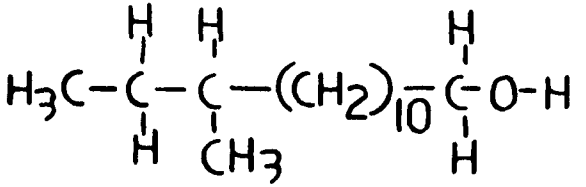
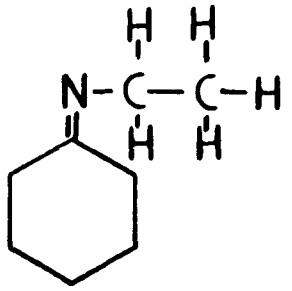
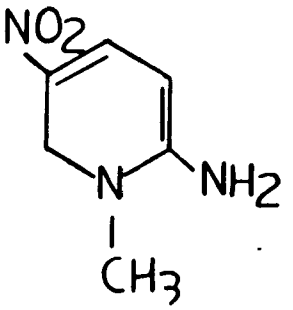
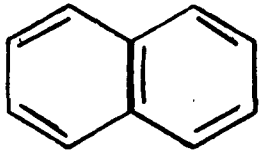
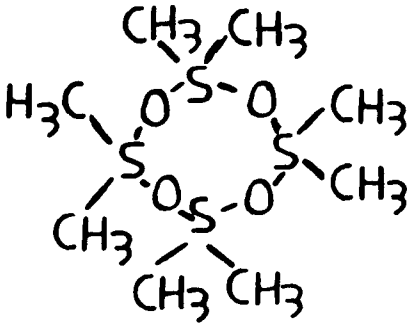
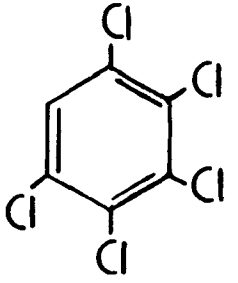
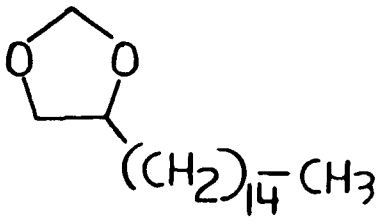
1. Benzene
2. Benzeneacetic acid
3. Benzonitrile
4. 3,5-bis (1,1-dimethylethyl)-1,2-Benzenediol
5. 3,4 bis [(Trimiehylisily)oxyl] - Estratrienone
6. Bromobenzene
7. 1-Bromo-2-Chlorobenzene
8. 5-Bromo-6-methyl-3-(1-methylpropyl)-Pyrimidinedione
9. Chlorobenzene
10. 4-Chloro-Benzonitrile
11. 3-Chloro-1,1'-Biphenyl-4-Ol
12. Chloro-4-(methylthio)-Benzene
13. 3-Chloro-2-Propenenitrile
14. Cyclohexane
15. Decamethyl-Cyclopentasiloxane
16. 1,2-Dichlorobenzene
17. 1,4-Dichlorobenzene
18. 2,6-Dichlorobenzonitrile
19. 4,7-Dichloro-benzo-2,1,3-Thiadiazole
20. 2,5-Dichloro-Thiazolopyrimidine
21. 5,7-Dichloro-Thiazolopyrimidine
22. 2,2-Dimethyl Hexane
23. Dodecamethyl-Cyclohexasiloxane
24. Ethyl-Indole carboxylic acid ethyl ester
25. Ethyl-methyl-Pyridinethione
26. Hexachlorobenzene
27. Hexachloro-1,3-Butadiene
28. Hexachlorodifluoro-Pentadiene
29. Hexamethyl Cyclotrisiloxane
30. Hexane
31. Hexanedioic acid, dioctyl ester
32. Isocyano-Naphthalene
33. 4-Methoxy-Benzoic acid Trimethylsilyl ester
34. Methylbenzene
35. 2-Methyl Benzofuran
36. 3-Methyl-2-Butanone
37. Methyl Cyclopentane
38. 12-methyl Tetradecanol
39. 12-methyl-1-Tetradecanol
40. N-ethyl-Cyclohexylamine
41. N-methyl-5-nitro-2-Pyridinamine
42. Naphthalene
43. Octamethyl Cyclotetrasiloxane
44. Pentachlorobenzene
45. Pentadecyl-1,3-Dioxolane
46. 2-Pentadecyl-1,3-Dioxolane
47. Silicate anion tetramer
48. Tetrachlorobenzene
49. 1,2,4,5-Tetrachlorobenzene
50. Tetrachloro-5-dichloromethylene-Cyclopentadiene
51. Tetrachloroethane
52. Tetramethyl Pentane
53. 2,4,6-Trichloro Benzenamine
54. 1,2,3-Trichlorobenzene
55. 1,3,5-Trichlorobenzene
56. 0,0,0-Tris-Trimethyl Epinephrine
57. 0,0,0-Tris-Trimethylisilyl Epinephrine

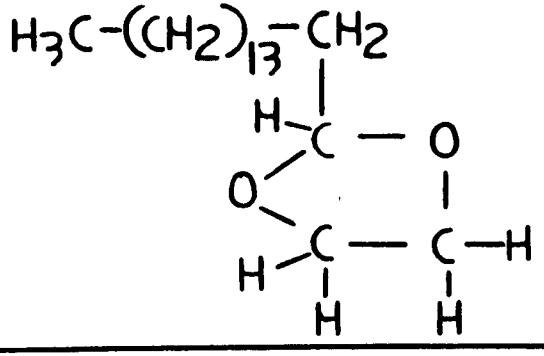
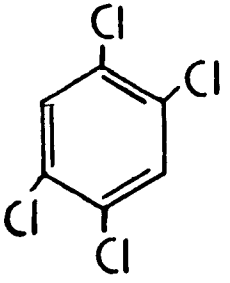
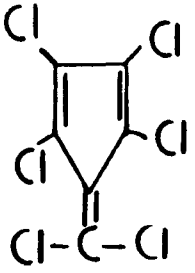
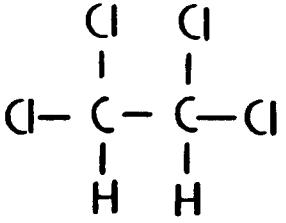
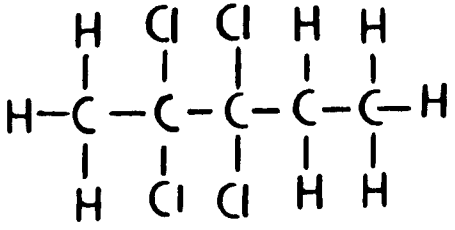
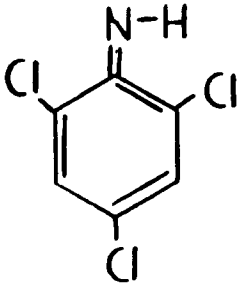
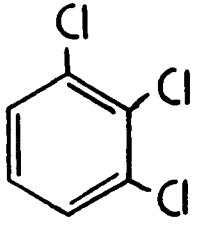
1 	**2** 	**3** 
4 	**5**  POSSIBE STRUCTURE	**6** 
7 	**8** 	**9** 

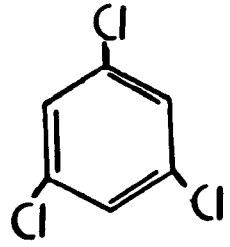
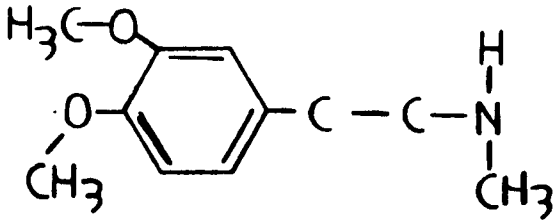
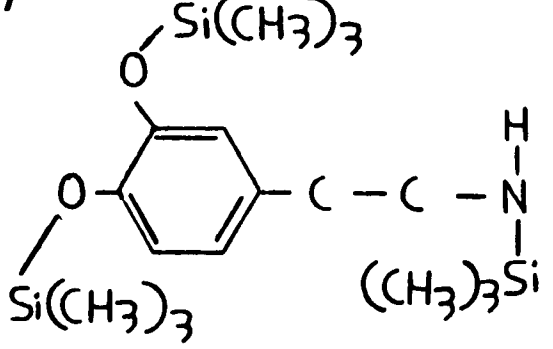
10 	**11** 	**12** 
13 	**14** 	**15** 
16 	**17** 	**18** 

19 	**20**	**21**
22 	**23** 	**24** 
25	**26** 	**27** 

28 	**29** 	**30** 
31 	**32** 	**33** 
34 	**35** 	**36** 

37 	**38** 	**39** SAME AS 38
40 	**41** 	**42** 
43 	**44** 	**45** 

46 	**47**	**48** 
49 SAME AS 48	**50** 	**51** 
52 	**53** 	**54** 

55 	**56** 	**57** 
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APPENDIX D

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LABORATORY TEST PLAN FOR INCINERATION OF BASIN F WASTES AT ROCKY MOUNTAIN ARSENAL

APRIL 1986

TASK NO. 17

CONTRACT NO DAAK11-84-D-0017

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1.0 TEST PLAN OVERVIEW

1.1 INTRODUCTION

Wastes in Basin F at Rocky Mountain Arsenal (RMA), which require treatment, include liquid and sludge as well as soils associated with the lagoon. These soils include fill, placed above and below the liner, as well as the 3/8-inch asphalt liner itself. These materials, which contain various concentrations of hazardous compounds as shown in Tables 1.1-1 and 1.1-2, are candidates for treatment by incineration. If treated by incineration, the hazardous organic compounds present in Basin F wastes must be destroyed at a destruction and removal efficiency (DRE) of 99.99 percent.

A conventional incinerator system, including an afterburner, subjects a compound to a variety of severe environments which may destroy hazardous waste at the desired DRE levels. Any organic compound subjected to hazardous waste incineration may be subjected to at least three, if not all, of the following environments:

1. Pyrolysis - Solids are volatilized or sublimed, volatiles and semivolatiles are evolved in the gas phase, and gaseous products may be further fragmented into smaller compounds and radicals;

2. Oxidation in the flame - Volatile compounds and radicals are subjected to a radical-rich environment and converted into CO_2, H_2O, and products of incomplete combustion (PICs);

3. Oxidation in a high temperature, postflame region - Final thermal reactions leading to complete oxidation of the organic constituents in the incinerator's combustion zone occur;

4. Oxidation in a second flame - Subsequent destruction (in the afterburner) of unreacted components of the hazardous wastes; and

TABLE 1.1-1

CHEMICAL CHARACTERIZATION OF BASIN F LIQUID

Compound or Parameter	Units	Concentration Range[1/]
pH	-	6.9 - 7.2
Aldrin	ppm	50.0 - 400
Isodrin	ppb	2.0 - 15
Dieldrin	ppb	5.0 - 110
Endrin	ppb	5.0 - 40
Dithiane	ppb	30.0 - 100
DIMP	ppm	10.0 - 20
DMMP	ppm	500.0 - 2,000
Sulfoxide	ppm	4.0 - 10
Sulfone	ppm	25.0 - 60
Chloride	ppm	48,000.0 - 56,000
Sulfate	ppm	21,000.0 - 25,000
Copper	ppm	700.0 - 750
Iron	ppm	5.0 - 6
Nitrogen	ppm	120.0 - 145
Phosphorus (total)	ppm	2,050.0 - 2,150
Hardness	ppm	2,100.0 - 2,800
Fluoride	ppm	110.0 - 117
Arsenic	ppm	1.0 - 1.3
Magnesium	ppm	35.0 - 40
Mercury	ppb	26.0 - 29
Cyanide	ppm	1.45 - 1.55
COD	ppm	24,500.0 - 26,000
TOC	ppm	20,500.0 - 22,500

1/ Based on analysis of various samples from different locations and depths in the basin (Buhts et al. 1977).

TABLE 1.1-2

HAZARDOUS CHEMICALS CONTAINED IN THE SOILS

VOLATILE ORGANICS

1,1-Dichloroethane	Benzene
Dichloromethane	Toluene
1,2-Dichloroethane	Xylene
1,1,1-Trichloroethane	Ethylbenzene
1,1,2-Trichloroethane	Chlorobenzene
Carbon tetrachloride	Methylisobutyl ketone
Chloroform	Dimethyldisulfide
Tetrachloroethylene	Bicycloheptadiene
Trichloroethylene	Dicyclopentadiene
Trans-1,2-Dichloroethylene	

SEMIVOLATILE ORGANICS

Aldrin	Oxathiane
Endrin	Dithiane
Dieldrin	Malathion
Isodrin	Parathion
p,p'-DDT	Chlordane
p,p'-DDE	Azodrin
Chlorophenylmethyl sulfide	Vapona
Chlorophenylmethyl sulfoxide	Supona
Chlorophenylmethyl sulfone	DIMP
Hexachlorocyclopentadiene	Atrazine

METALS

Aluminum	Iron
Arsenic	Lead
Cadmium	Mercury
Chromium	Zinc
Copper	

5. Oxidation in a second high temperature, postflame region - Final thermal oxidation reactions occur before the combustion gases are exhausted to pollution control devices.

In general, it is believed that 99 percent of the destruction of any hazardous organic compound occurs in the flame region. The postflame region destroys 99 percent of the remaining one percent of material to achieve the 99.99 percent destruction removal efficiency (DRE). (For detailed discussions, see Kramlich et al. 1984, and Dellinger et al. 1984.) Pyrolysis reactions are shown in Figure 1.1-1. Flame and postflame reactions are summarized in Figure 1.1-1 as "Thermal Oxidation."

The bench-scale laboratory program, designed and described in the following pages, recognizes the difficulty in handling the compounds listed in Tables 1.1-1 and 1.1-2 and the complexity of thermal destruction through incineration as illustrated in Figure 1.1-1. This program is designed, therefore, to accomplish the following:

1. Provide sufficient information on the physical, chemical, and thermodynamic properties of the compounds listed in Table 1.1-1 and 1.1-2 to ensure reasonable success in designing and implementing an incineration program;

2. Provide a bench-scale apparatus that accurately simulates all or a major portion of a full-scale incineration system;

3. Demonstrate the achievement or potential to achieve 99.99 percent DRE for hazardous compounds present in Basin F; and

4. Contribute to the selection of an incineration technology.

Data developed by Dellinger et al. (1984) demonstrate that most organics can be incinerated to a DRE of 99.99 percent within two seconds at temperatures of 600°C to 950°C (Table 1.1-3). In addition, Kramlich et al. (1984), have determined that excess air used in the

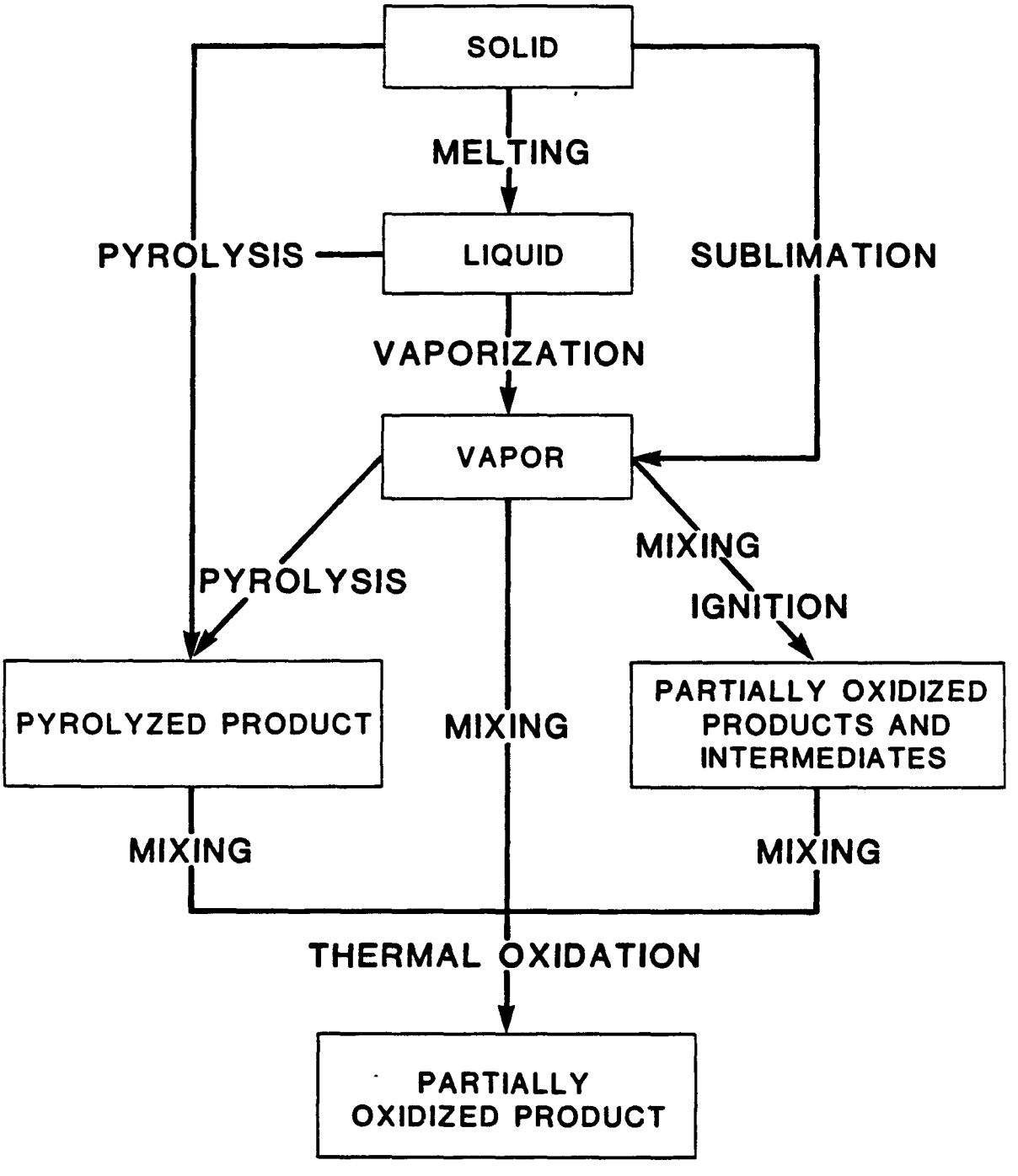

FIGURE 1.1-1

SCHEMATIC DIAGRAM OF PROCESSES OCCURRING DURING THE DESTRUCTION OF A SOLID WASTE

flame mode of destruction is best held within 30 percent to 40 percent (35 percent excess air corresponds to 5.4 percent O_2 in the dry stack gas) to produce the lowest levels of CO and hydrocarbon emissions, and ensure the most complete combustion of any supplementary fuel, as well as hazardous wastes. Consequently, the bench-scale program has been designed recognizing the fairly narrow ranges of temperatures, times, and excess air associated with achieving 99.99 percent DRE of most organic hazardous wastes.

1.2 LABORATORY TEST PROGRAM OBJECTIVES

The laboratory test program has been designed to accomplish the following objectives:

- Demonstrate that 99.99 percent DRE is achievable for the hazardous wastes contained in the liquid, sludge, and contaminated soils associated with Basin F;

- Determine what temperatures, residence times, and levels of excess O_2 can be used to achieve 99.99 percent DRE within the most cost-effective incinerator technology framework;

- Provide sufficient data to determine hazardous waste destruction kinetics based on first order approximations;

- Provide guidance for final incineration technology selection and optimization for transition from bench-scale to pilot plant or from bench-scale to a full-scale operation. In this respect, bench-scale testing is designed to provide guidance for initial conditions and subsequent conditions to be tested by the next scale of operation.

TABLE 1.1-3

SUMMARY OF THERMAL DECOMPOSITION DATA

Compound	Empirical Formula	T_{onset}(1) (°C)	T_{99}(2) (°C)	$T_{99.99}$(2) (°C)
Acetonitrile	C_2H_3N	760	900	950
Tetrachloroethylene	C_2Cl_4	660	850	920
Acrylonitrile	C_3H_3N	650	830	860
Methane	CH_4	660	830	870
Hexachlorobenzene	C_6Cl_6	650	820	880
1,2,3,4-Tetrachlorobenzene	$C_6H_2Cl_4$	660	800	850
Pyridine	C_5H_5N	620	770	840
Dichloromethane	CH_2Cl_2	650	770	780
Carbon Tetrachloride	CCl_4	600	750	820
Hexachlorobutadiene	C_4Cl_6	620	750	780
1,2,4-Trichlorobenzene	$C_6H_3Cl_3$	640	750	790
1,2-Dichlorobenzene	$C_6H_4Cl_2$	630	740	780
Ethane	C_2H_6	500	735	785
Benzene	C_6H_6	630	730	760
Aniline	C_6H_7N	620	730	750
Monochlorobenzene	C_6H_5Cl	540	710	780
Nitrobenzene	$C_6H_5NO_2$	570	670	700
Hexachlorethane	C_2Cl_6	470	600	640
Chloroform	$CHCl_3$	410	590	620
1,1,1-Trichlorethane	$C_2H_3Cl_3$	390	570	600

1/ Temperature at which decomposition initiates at 2 seconds reaction time.

2/ Temperature where 99 and 99.99% of the compound is destroyed at a 2 second reaction time.

Source: Dellinger et al. 1984.

1.3 TECHNICAL APPROACH OVERVIEW

Given the nature of the compounds being destroyed and the objectives of the program, a technical approach has been developed to ensure success of the ultimate full-scale incineration effort. This approach recognizes the inherent limitations of laboratory investigations, along with the lack of precise data concerning the feedstocks to be incinerated.

The technical approach involves using equipment that will simulate three of the major incineration mechanisms: 1) pyrolysis; 2) postflame (primary incinerator); and 3) postflame (afterburner). Basin F samples will be sent to Hittman/Ebasaco Associates Inc. (HEAI) for preparation. HEAI will perform the actual bench-scale incineration testing. If necessary, HEAI will send the feed samples to UBTL and CAL for laboratory analysis. The tests will be carried out with the largest sample sizes possible given the constraints of laboratory operations in order to ensure data accuracy in scale-up. Large-scale (e.g., 250-500 gram) samples are to be used. Because relatively large samples are being used, testing of the consequences of flame-mode destruction cannot be directly simulated.

The technical approach is designed largely to focus on and evaluate the impact of incineration on Basin F contaminated soil. The incineration regime found to be successful with soil then will be confirmed for the incineration of liquid and sludge. This approach, initially, does not designate one or more principal organic hazardous constituents (POHCs), but evaluates the impact of incineration on all compounds identified in Tables 1.1-1 and 1.1-2.

The technical approach begins with limited characterization of selected compounds in terms of physical, chemical, and thermodynamic properties. Of most significance are the ash fusion temperatures of the principle types of soil and, consequently, the potential for operating any incineration system in the slagging mode. The technical approach then tests the impact of incineration of contaminated soil at two temperatures, two residence times (in the afterburner), and two levels of excess O_2. Multiple runs will be used to ensure that the DRE associated with any compound will not be masked,

regardless of concentration in the incoming material to be incinerated. The impact of incineration on sludge, liquid, and a proportionate mixture of liquid, sludge, and soil will be the final test sequence.

The actual matrix of test conditions is summarized below:

Parameter	Maximum Value	Second Value	Minimum Value
Temperature	1,250°C	900°C	650°C
Time	5 sec	2 sec	N/A
O_2 Level	5.4%	7.0%	N/A

This rationale, discussed in Chapter 4, is based upon conditions expected to occur in a full-scale incinerator system. Further, this rationale is based upon providing sufficient spread in the parameters to permit extrapolation of results between extreme points.

1.4 EXPECTED RESULTS

Test program results will facilitate scale-up of the bench-scale thermal destruction system to either pilot plant or full-scale operations. Expected results include the following specific data:

- Evaluation of hazardous chemicals remaining in the residues of soils or sludges after incineration;
- Degree of destruction associated with specific pyrolysis and postflame environments, to determine acceptable regimes for incineration processes (e.g., temperature in the afterburner, residence time, and excess O_2 in the flue gas); and
- Sufficient time, temperature, and oxygen concentration data to extrapolate rough optimal conditions between the tested points identified above, assuming first order kinetics.

The bench-scale program, designed to test for the destruction of all hazardous waste compounds identified in Tables 1.1-1 and 1.1-2, will permit final selection of POHCs to be used for determination of the success of larger-scale systems. Additionally, pretesting of soil, sludge, liquid, and selected hazardous materials will provide essential physical, chemical, and thermodynamic data for design and operation of the primary thermal destruction (incinerator) unit at either the pilot-scale or full-scale. These data will include, but not necessarily be limited to:

- o Ash fusion temperature of the soil;
- o Thermal conductivity, specific heat, and heat capacity of the soil and sludge;
- o Selected calorific values, proximate analyses, and related data for the compounds to be incinerated; and
- o Corrosivity (with particular respect to refractories) of the liquid with five percent chlorides and two percent sulfides.

These data will assist in the determination of fuel requirements, residence times of solids, and desired temperatures associated with the pilot plant and full-scale primary incinerator. They also will be used to determine the maximum temperature associated with the Linder furnace in the bench-scale test. The Linder furnace accomplishes the solids' heatup and volatile evolution. While full-scale operation may occur in the slagging mode, the bench-scale apparatus will be operated below slagging temperatures.

The ultimate value of the bench-scale test program will be to develop data for operation scale-up. The results described above will assist not only in determination of a combustion regime that will achieve 99.99 percent DRE, but also will confirm the most appropriate technology for incineration of Basin F waste. Specific parameters associated with technology selection will be temperatures, the evolution of hazardous chemicals from soil and sludge, residence times, and excess oxygen levels. Specific technologies

to which these data can be applied include countercurrent and cocurrent rotary kilns, fluidized beds, and hearth-type furnaces.

The bench scale test regimes have been designed to achieve 99.99 percent DRE. The most severe conditions including a temperature of 1250°C and a residence time of 5 sec exceed those used by other researchers (see, for example, Dellinger et al. 1984) to achieve 99.99 percent DRE. The margins of safety to ensure that the desired DRE is obtained exist both in temperature and time. These margins of safety have been selected based on a review of the literature associated with hazardous waste destruction, where the hazardous compounds are in dilute concentrations.

It is recognized, however, that the laboratory program simulates the post-flame oxidation zone, but does not simulate flame-mode destruction of hazardous chemicals. The laboratory test program, then, does not simulate the most severe environments available. Such environments include higher temperatures, if shorter residence times. Typical flame temperatures may be about 1725°C (2000 K), and residence times may be 0.1 sec (Perry et al. 1963). Further, the flame environment is characterized by high concentrations of free radicals; and consequently the mechanisms for hazardous waste destruction in that environment are most different from those associated with the post-flame oxidation zone. Radical dominated mechanisms increase the rate of hazardous waste destruction relative to that rate associated with oxygen-rich non-flame environments (Kramlich et al. 1984). Consequently, the destruction in an incinerator typically occurs as follows: 1) 99 percent within the flame region and 2) 99 percent of the remaining 1 percent of material in the post-flame region.

Given these data, it is reasonable to conclude that the laboratory program, by itself, will achieve 99.99 percent DRE levels for the wastes in Basin F. Dilute concentration kinetics as developed by previous research leads to this conclusion. At the same time, however, the experiment is not simulating the flame mode destruction. Consequently, it is not simulating one mechanism for achieving at least 99 percent DRE. Given that consideration, if the laboratory test achieves in excess of 99 percent DRE, it is reasonable to conclude that a pilot plant or full-scale incinerator would achieve 99.99 percent DRE when combining flame mode destruction with a strong post-flame oxygen-rich environment.

2.0 BENCH-SCALE INCINERATION

2.1 BENCH-SCALE TEST SYSTEM

2.1.1 Rationale for a Bench-Scale System

Thermal decomposition laboratory tests have been performed on both pure compounds and field samples to determine incineration parameters, including temperature, residence time, and excess oxygen, required to decompose toxic chemicals. These laboratory tests have been performed, primarily, using milligram-to-gram size samples. These small sample sizes have been adequate to characterize incineration parameters for pure compounds and compounds in high concentrations. For chemicals which are present in low concentrations, these small sample sizes are not adequate to demonstrate 99.99% destruction due to the analytical limits of detection of the off-gases. It is of interest to demonstrate 99.99% destruction for all toxic constituents in a feed sample regardless of whether or not that constituent is chosen as a principal organic hazardous constituent (POHC). Although there are substantial data on the thermal destruction of individual compounds, incineration tests on field samples are necessary to adequately simulate the interaction of various constituents at high temperatures and the production of products of incomplete combustion (PIC). The bench-scale test unit for Task 17 was designed to measure DRE up to 99.99% for constituents of concern at Basin F for soil, sludge, and liquid samples.

2.1.2 Design Philosophy

The laboratory bench-scale unit was designed to evaluate thermal destruction efficiency at temperatures up to 1300°C and residence times from 2 to 5 seconds. The unit is a batch-load system with two furnaces and a blended carrier gas to simulate combustion gases (Figure 2.1-1). The first furnace is used to volatilize the constituents. The carrier gas moves these constituents into the secondary furnace which is used

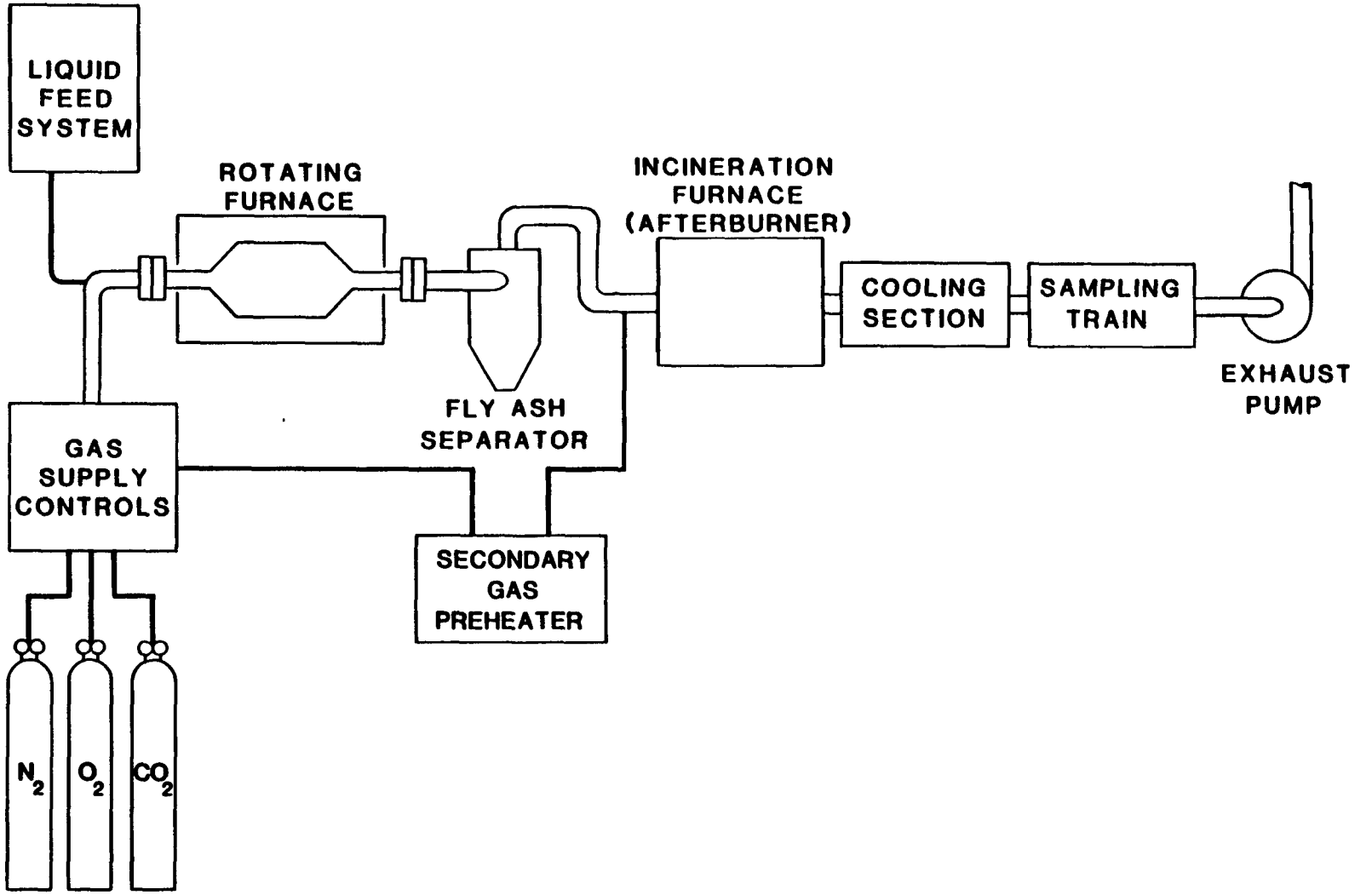

FIGURE 2.1-1

LABORATORY SCALE INCINERATION UNIT

to simulate afterburners in a full-scale incineration plant. In the secondary furnace, additional blended gases with O_2 are added and temperature is increased to decompose the hazardous constituents. The combustion products in the off-gas are then collected in various sorbents in the sampling train.

2.1.3 Sample Size

The first design consideration was that of the overall apparatus size. The primary concern with respect to this was that of being able to collect and analyze an off-gas constituent to demonstrate 99.99% DRE of a chemical present in the feed sample in a few parts per billion. For the chlorinated compounds which can be analyzed using GC/ECD, a sample size of several hundred grams is adequate. Task budget and availability of a rotating tube furnace, capable of handling batch samples up to five hundred grams, determined the laboratory bench-scale unit design based on a sample size of 200-500 grams.

2.1.4 Primary Furnace

The primary furnace (Figure 2.1-2) is an electric furnace with a programmable temperature controller capable of maintaining 1000°C with gas flows up to 20 liters per minute. A gas supply system is used to provide blends of N_2, CO_2, and O_2 to simulate various combustion processes in fuels. The primary furnace barrel (Figure 2.1-3) is 130 mm in diameter and 200 mm in length. The maximum temperature rise of the primary furnace is about 5.5°C per minute. The carrier gas velocity will be between 6 and 8 cm per second at test conditions.

2.1.5 Fly Ash Trap

Provisions will be made for a fly ash separator between the primary and secondary furnace. The purpose of this separator is to remove ash which may be entrained in the carrier gas and to prevent plugging of the secondary furnace. The ash separator will be a cyclone type design

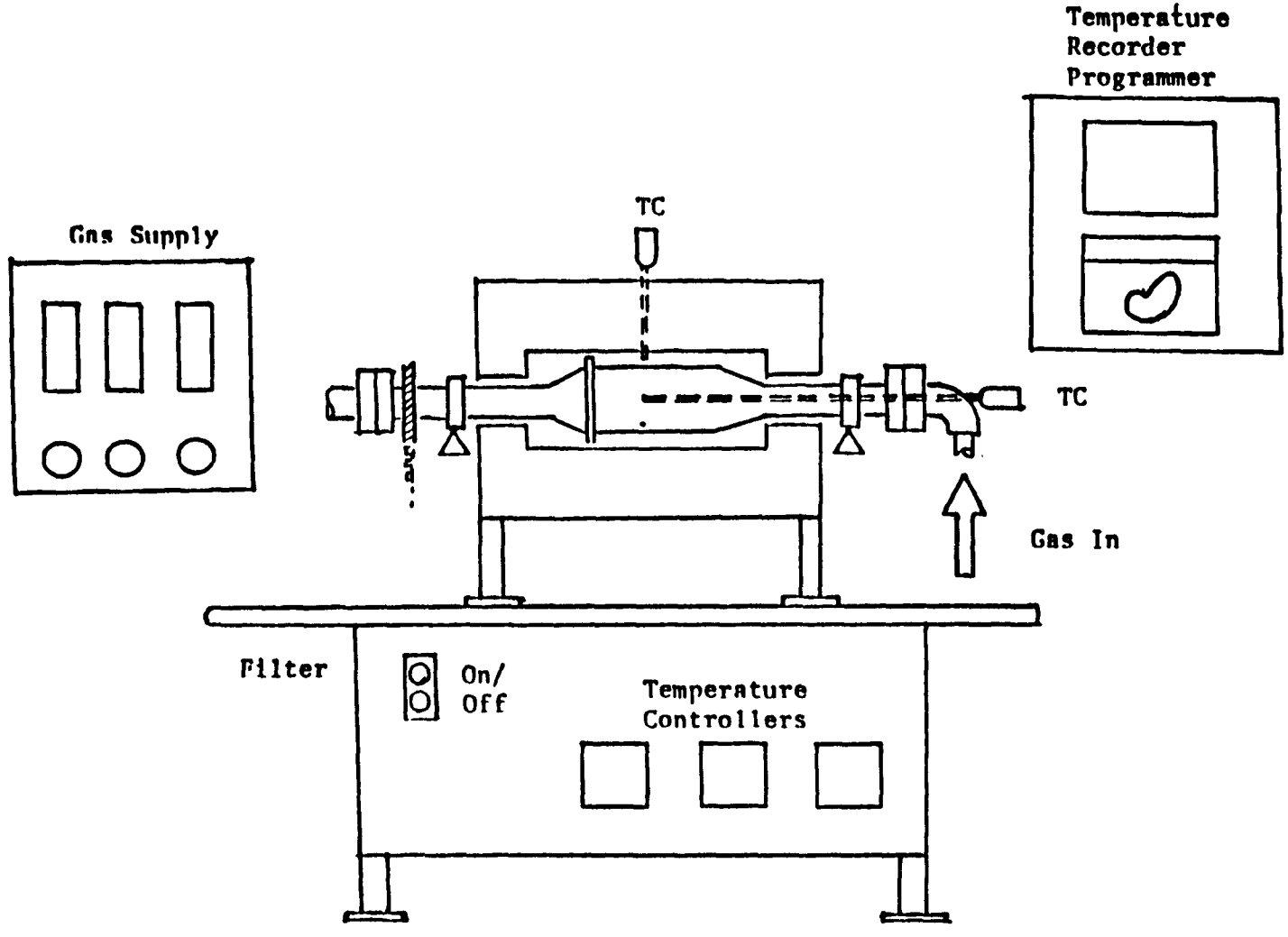

FIGURE 2.1-2
ROTATING TUBE FURNACE ARRANGEMENT

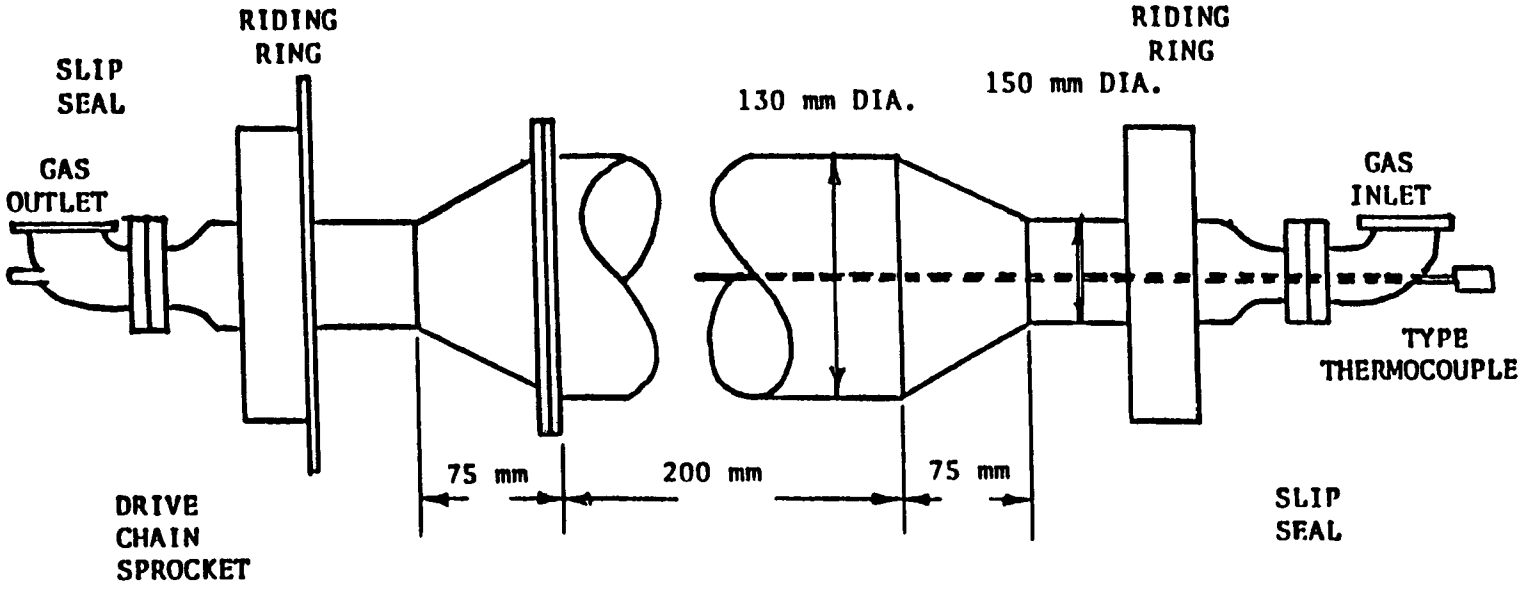

FIGURE 2.1-3
ROTATING TUBE UNIT

capable of removing particulates down to 100 microns. It will be constructed of stainless steel and insulated to prevent heat loss between the primary and secondary furnaces.

2.1.6 Secondary Combustion Gas

Additional gases will be introduced between the primary and secondary furnaces to simulate secondary combustion gases. The composition of this gas will be the same as that of the primary carrier gas and will increase the total gas flow rate by 50 percent. The carrier gas will be preheated to near (± 50 C°) that of the primary carrier gas temperature.

2.1.7 Secondary Furnace

The secondary furnace was designed to heat gases from the primary furnace along with the secondary airflow up to 1250°C and to maintain the gases at this temperature for between 2 and 5 seconds. To have fully developed flow while avoiding high pressure losses in the furnance, a velocity range of 20 cm/sec to 500 cm/sec was established. For this velocity range and the desired gas flow rate, the furnace tube diameter would be approximately 2 1/2 cm. For a residence time of 5 seconds, the furnace tube would be approximately 10 meters long. The secondary furnace tube will be constructed of fused quartz to provide a nonreactive environment at high temperature. With proper bending of the quartz tube, the secondary furnace would require a 0.14-cubic meter volume. This size is consistent with small pottery kilns which are capable of withstanding a temperature of up to 1300°C. Three temperature probes will be installed in the secondary furnace to monitor furnace temperature. The kiln should be able to maintain temperature within ± 10 C°.

Gas residence time in the secondary furnace can be varied by changing gas flow rate or length of the furnace tube. Since the desired residence times range from 2 to 5 seconds, the gas flow rate would have

to be varied by a factor of 2.5 to cover that range. Since this variation in the gas flow rate is too large to maintain reasonably consistent test conditions, two furnace tube lengths will be used.

2.1.8 Cooling Section

The cooling section will consist of a straight 2.5-cm diameter quartz tube approximately 3 feet long. Exit temperature from the cooling section will be monitored to insure that temperature will be maintained between 200°C and 300°C. Insulation will be applied to the tube to adjust the exit temperature.

2.1.9 Sample Collection

All off-gases from the secondary furnace will enter the sample collection system. The sample collection system is designed to remove organic and inorganic constituents of concern. A pump will be used downstream of the sample collection system to maintain a near atmospheric pressure in the entire flow train. The sample collection system is described in detail in Section 2.2.

2.1.10 Range of Test Conditions

Once the design of the bench-scale test apparatus is fixed, variations in test conditions from the design point are possible; however, parameters are interdependent for a fixed furnace volume. Residence time in the furnace is a function of volume flow rate which is a function of the mass flow rate, pressure, and temperature (Figures 2.1-4 and 2.1-5). Actual operating regimes will be established as the design is finalized.

2.2 SAMPLE COLLECTION SYSTEM

Collection of the gases generated from all test incineration runs will require a system for collection of non-particulate and particulate

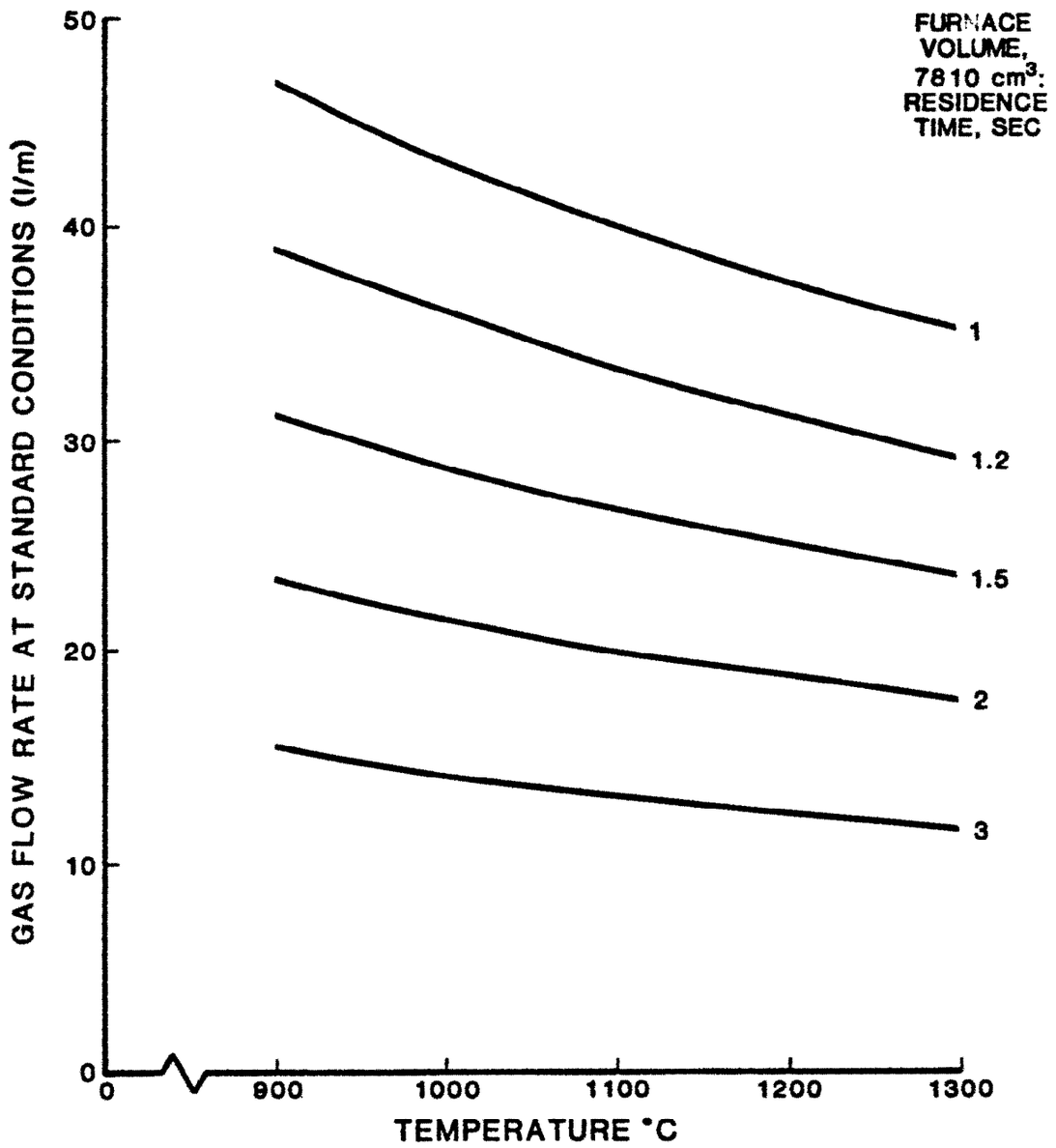

FIGURE 2.1-4
TEST CONDITION RANGE

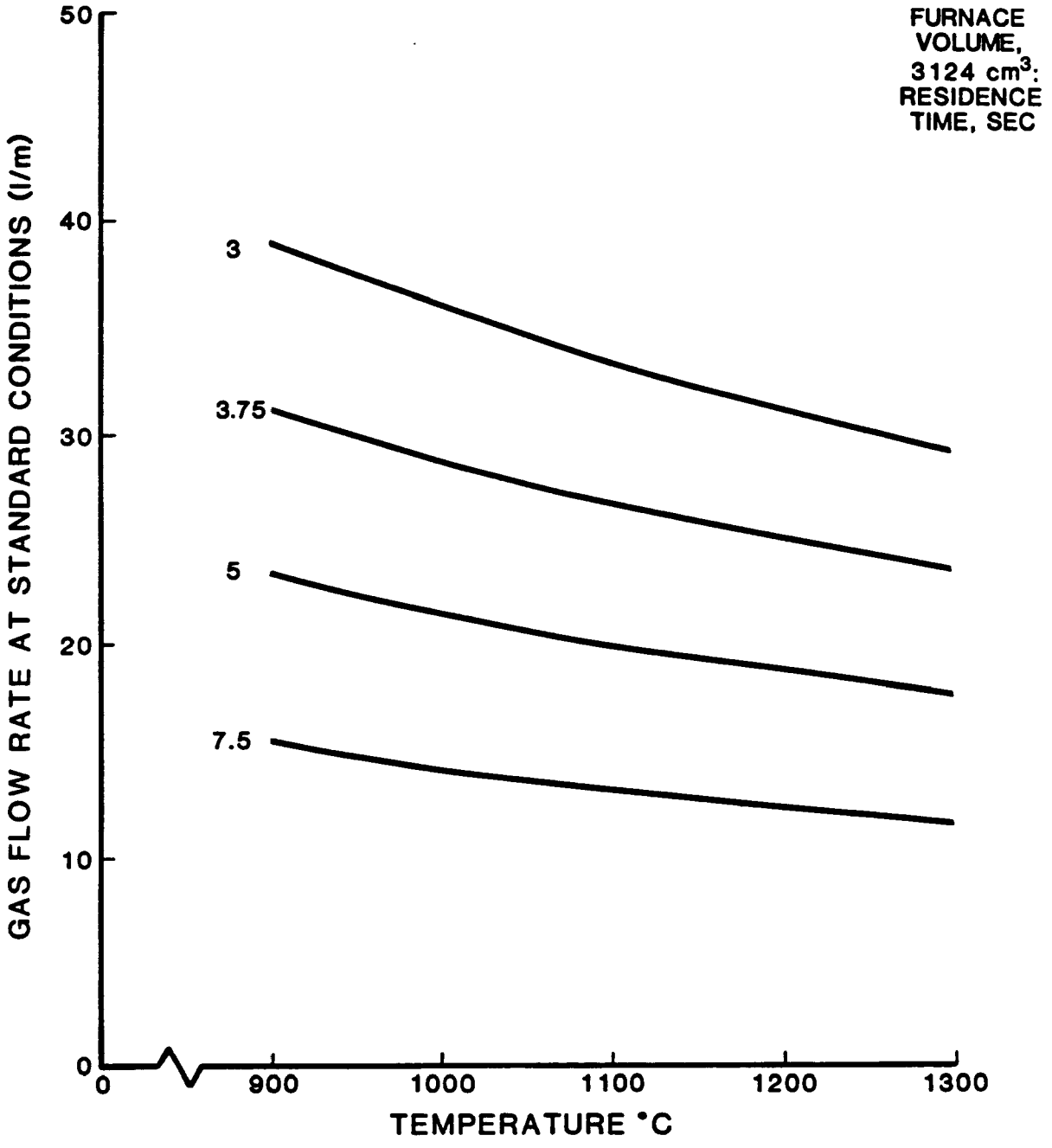

FIGURE 2.1-5

TEST CONDITION RANGE

fractions. Sampling of off-gases depends on the nature of POHCs and other large species. In general, the sampling apparatus for collecting off-gas effluents includes three major components:

- o One or more thermostatically controlled compartments to maintain the gas at a temperature consistent with the collection medium, usually hot (200°C) for particulate collection and cool (20°C) for sorbent collection of the more volatile constituents;

- o Sample collectors to collect the samples, such as, filters and sorbents; and

- o Vacuum pump and gas meter

The sampling train used will be similar to the one shown in Figure 2.2-1. Using this sampling train will provide both adequate trapping of particulate and non-particulate fractions from the off-gas. The number of impingers and sorbent tubes may vary in number and type depending upon the test run. This device is physically similar to the Modified Method 5 (MM5) sampling train.

2.2.1 Particulate and Residue Collection

Bottom residue left in the kiln from the test burn will be removed by the most efficient means available to the lab which will be consistent with:

- o Complete removal (>99%);
- o Prevention of outside contamination; and
- o Prevention of damage to the kiln.

Bottom residue removal will be dependent upon the physical characteristic of the test sample after incineration. The bottom residue mass may vary from <10% of the starting material weight to >90% depending upon the sample matrix (liquid, sludge, soil). The laboratory anticipates some flexibility will be required in attaining

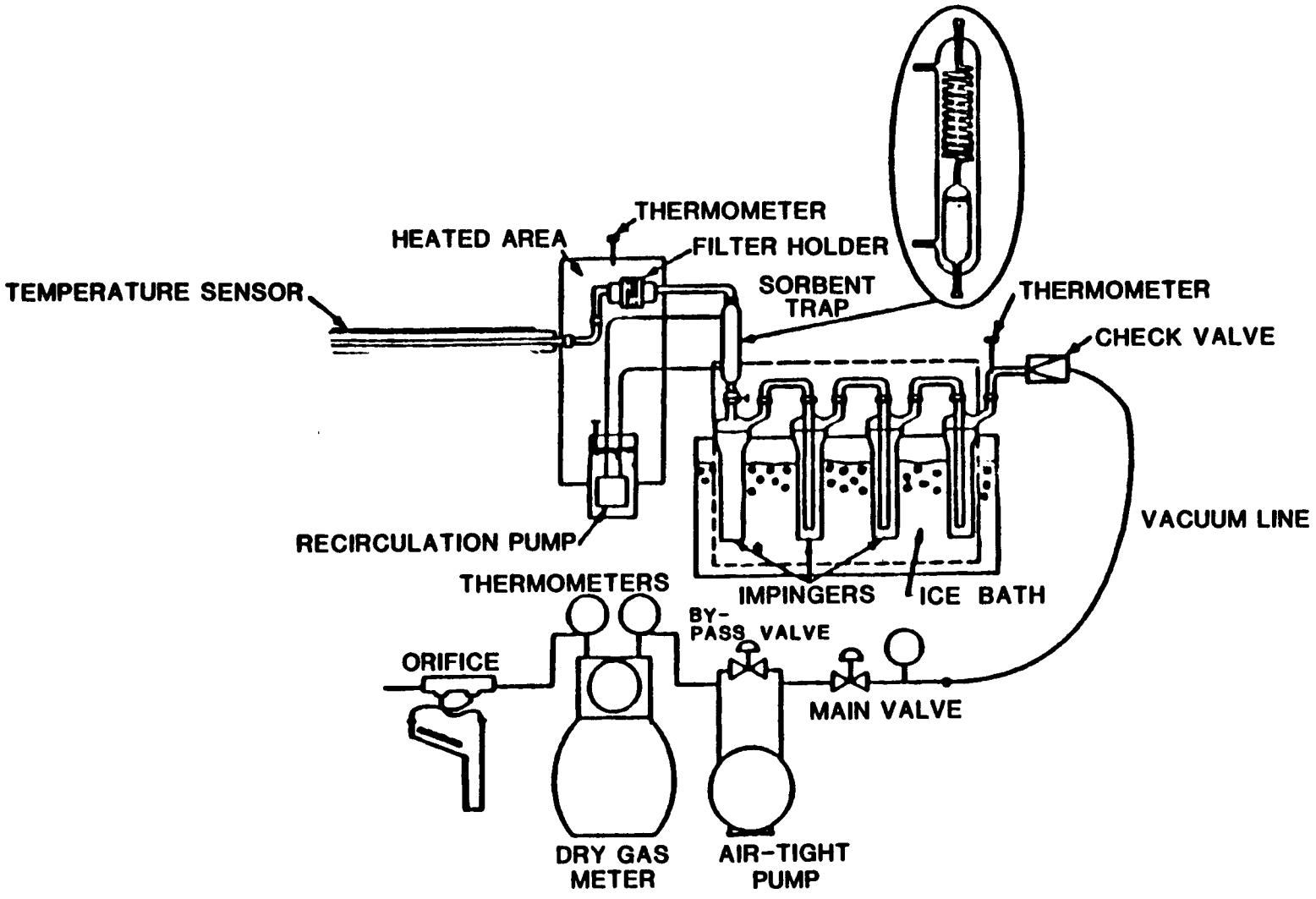

FIGURE 2.2-1
SAMPLE TRAIN

an efficient removal of the bottom residue. Residue removal and cleaning of the kiln will be adequate to assure subsequent test burns are not cross-contaminated. Bottom residue will be stored at about 4°C in glass bottles with Teflon lined caps until combined with the fly ash.

The fly ash separator will retain the larger particulates carried through the primary furnace tube. As with the bottom residue, the volume of fly ash produced will vary with respect to sample matrix. Efficient removal of the fly ash to Teflon-capped glass bottles can be expected. The fly ash will be stored at about 4°C.

Filter cassettes will be used to trap particulates which are not separated as fly ash and may vary in size from 1 to 100 microns. The filter used will be a glass fiber type and will be stored in a glass bottle with a Teflon-lined cap at 4°C.

Figure 2.2-2 illustrates the flow of the residue sample into the analytical system. The three solid fractions from the test burns are weighed and the weight summed to estimate the percentage of sample volatilized:

$$\% \text{ Sample Volatilized} = \left(1 - \frac{W_B + W_F + W_P}{W_S}\right) \times 100$$

Where W_B = Weight of Bottom Residue
W_F = Weight of Fly Ash
W_P = Weight of Filter Particulates
W_S = Weight of Original Sample

The bottom ash and fly ash will be combined and homogenized. Aliquots of this residue will be taken for the various chemical and physical analyses required to determine destruction efficiency of the POHCs and the EP toxicity of the residue.

The particulate filter is weighed and combined with the XAD-2 resin for extraction and analysis.

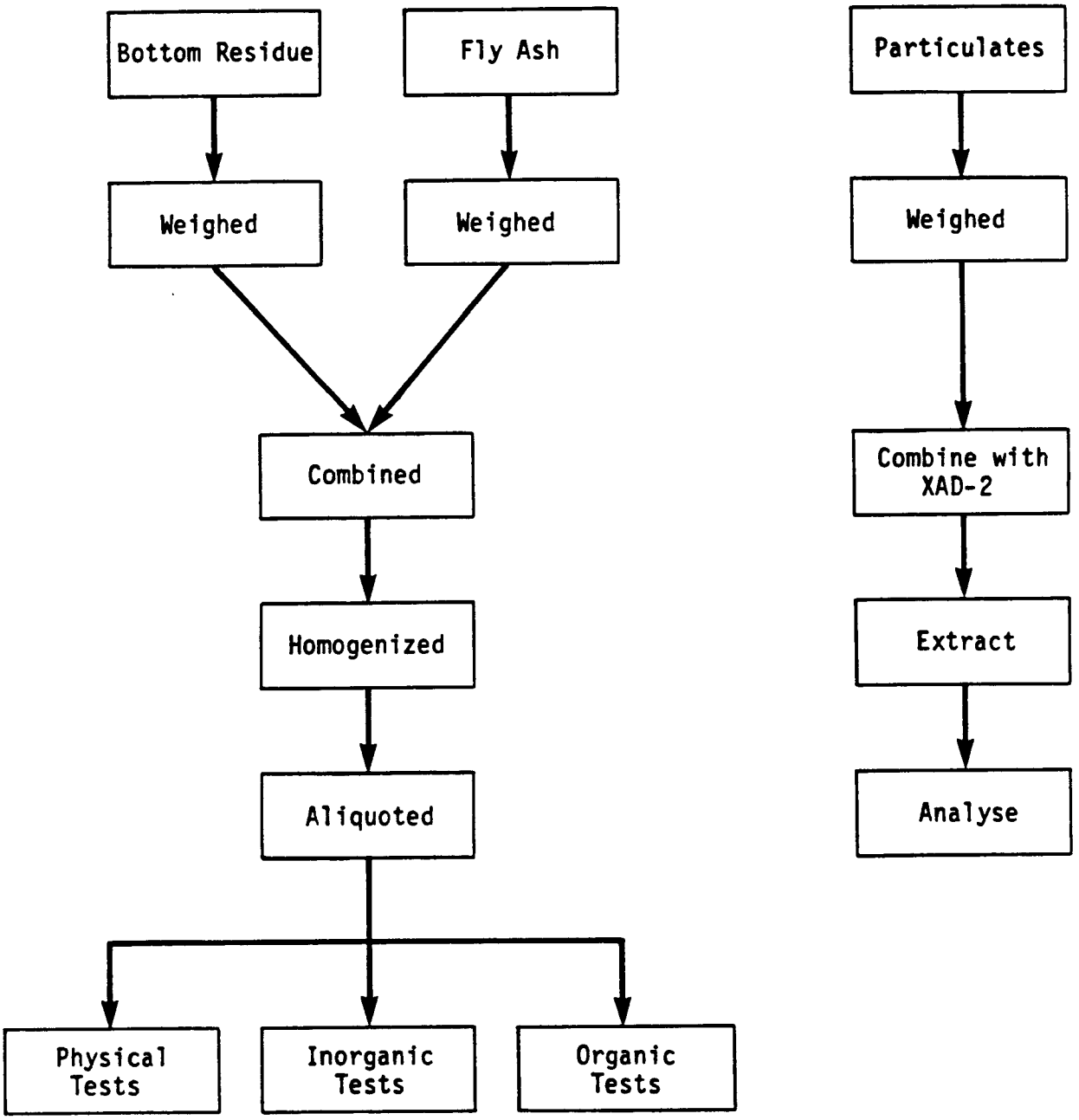

FIGURE 2.2-2

SOLID RESIDUE COLLECTION FLOW CHART

The chemical and physical analyses to be performed on these incineration residues are described in detail in Section 5.0. Section 4.3 contains details of the analytical test matrix after the incineration tests.

2.2.2 Gas Collection

The gas collection procedures are dependent upon the POHCs that have been selected for analysis to determine if they have been destroyed to 99.99% DRE. The PICs are also important in the selection of the types of impingers and sorbents used. As a result of the initial sample size, it is necessary to completely extract and concentrate the XAD-2 sorbent, and combine it with the extracted condensate, and the other impinger solutions to achieve parts per trillion detection limits.

Table 2.2-1 describes the types of sorbent and impinger solutions that will be used to trap organic and inorganic products from the incineration. When the waste sample matrix is water or sludge, a condensate trap will be used to reduce the volume of liquids delivered to the sorbent traps. Figure 2.2-3 describes this trap. The condensate collected in the trap must be tested for the various compound classes. An aliquot of the liquid can be analyzed for volatile and semivolatile organics and acid and basic inorganics (i.e., F^-, Cl^-, phosphorous, and metals). Section 5.2 provides more details on the analytical protocol for handling the condensate fraction.

After a test run, the sorbents and impinger fractions, as well as the condensate when applicable, are transferred to glass bottles with Teflon-lined caps for storage at about 4°C. The analytical protocols which can be performed on the various fractions are described in Section 5.0.

TABLE 2.2-1

GAS SAMPLE COLLECTION MATRIX

Compound Class	Sorbent	Impinger	Water * Trap
Volatile Organics	Tenax/Charcoal		Test
Semivolatile Organics	XAD-2		Test
Volatile Metals		Silver Catalyzed Ammonia Persulfate	Test
Acid Compounds		0.1 NaOH	Test
Cyanide		0.1 NaOH	Test
Basic Compounds		0.1 HCl	Test

*A water trap will be utilized when the test sample is sludge or liquid. (See text.)

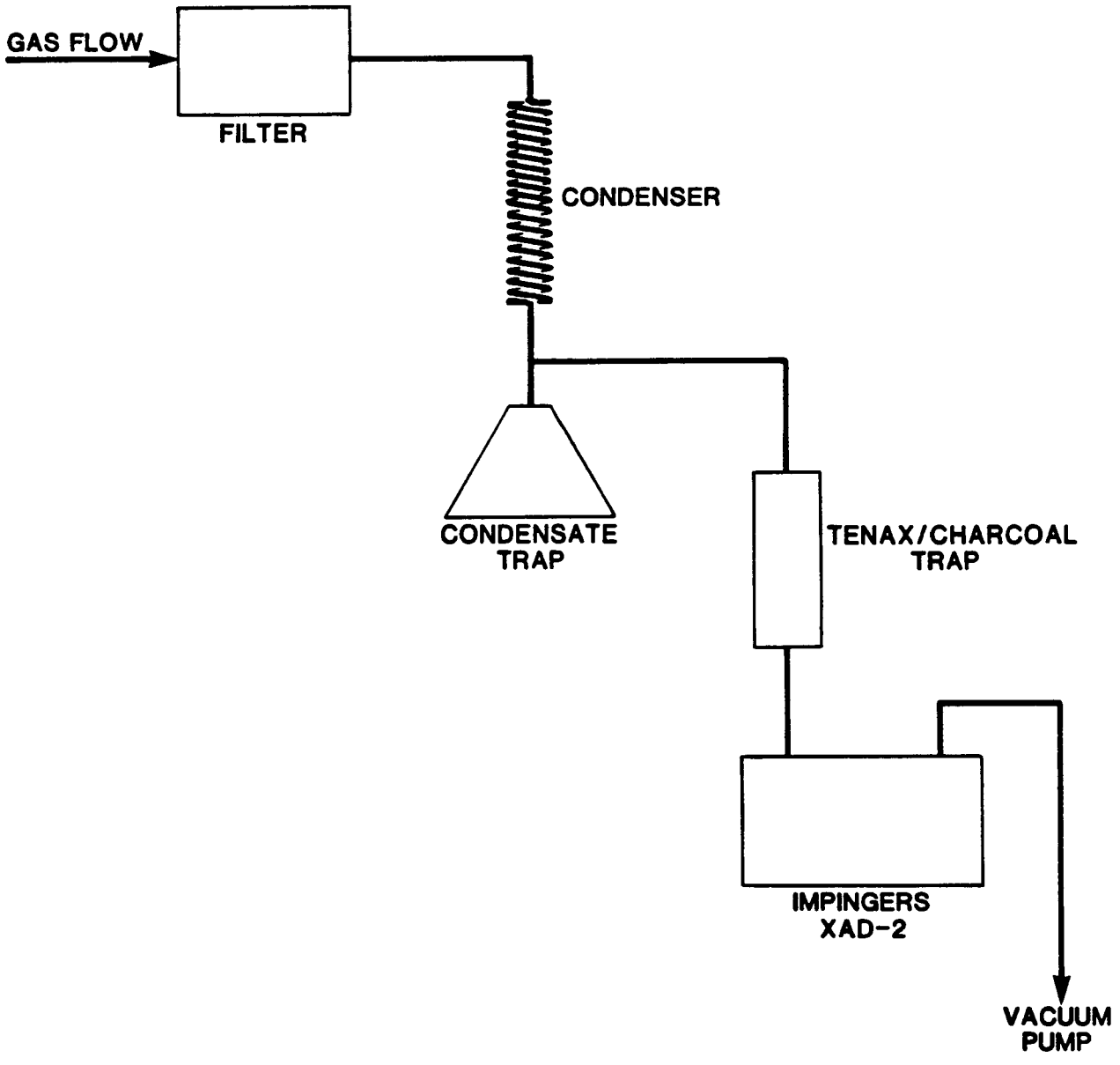

FIGURE 2.2-3
MODIFIED SAMPLING TRAIN FOR HIGH MOISTURE SAMPLES

2.3 BENCH-SCALE TEST OPERATIONS

A detailed operating procedure will be developed after the final design of the bench-scale unit and modified during the course of the system checkout. The following sections outline some of the operational considerations for the soil, sludge, and liquid tests.

2.3.1 Soil Tests

Typical operation of the bench-scale test aparatus for soil samples will involve the following:

Step

1. Weigh out appropriate sample size (200-500 grams $\pm$ 0.5 grams).

2. Place the sample in the kiln barrel and bolt the barrel halves together.

3. Place the kiln barrel into the furnace and attach the thermocouple and gas connections.

4. Set the secondary furnace temperature and allow it to reach test condition temperature before proceeding.

5. Switch on the evacuation exhaust pump.

6. Establish carrier gas flow at the desired blend and flow rate.

7. Start temperature ramp on primary furnace.

8. After reaching the desired test temperature on the primary furnace, start barrel rotation and maintain desired test conditions for one hour before starting shut down procedures.

9. Turn primary furnace off and stop barrel rotation, but continue gas flow.

10. After primary furnace has cooled to 400°C, turn off secondary furnace.

11. Divert gas from sampling train and remove collected samples.

12. After primary furnace has cooled to near room temperature, remove kiln barrel and disassemble.

13. Remove residual sample from barrel.

14. Disassemble fly ash collection system and remove fly ash.

15. During the course of the system operation, the following parameters will be monitored and recorded: N_2, CO_2 and O_2 flow rate of primary and secondary gasses, temperature of the rotating kiln gas, fly ash separation system exit gas, secondary furnace, and cooling section exit gas, particulate sample isothermal box and impinger isothermal box. Sample train flow meter delta pressure also will be monitored.

2.3.2 Sludge Tests

The operation of the bench-scale apparatus during sludge tests would be the same as that for the soil tests except for those considerations necessary to deal with the high moisture content of the sample. The following additions or changes would be made to the operating procedure:

Step

1-6. Identical to soil tests (2.3.1).

7. The primary furnace temperature will be raised to 90°C and held at this temperature until most of the moisture is removed from the sample. The carrier gas flow rates will be reduced during this drying period to compensate for the increased flow rate due

to the water vapor. This adjustment is necessary to maintain the desired residence time of the gases through the secondary furnace.

8-15. Identical to Soil Tests (2.3.1).

(Note: A condensate trap will be placed between the particulate filter and the sorbant traps in the sampling train to remove the high load of moisture. The moisture in the trap will be analyzed for POHCs).

2.3.3 Liquid Tests

Unlike the soils and sludges which would be batch fed, the liquid waste would be continuously fed through a probe into the primary furnace barrel. The desired temperatures and carrier gas flows would be established in both the primary and secondary furnace prior to feeding the liquid waste. The liquid waste systems would consist of a reservoir and peristaltic pump. For a 300-gram liquid sample fed into the primary furnace over a 1-hour period, the sample volume flow rate in the secondary furnace would be approximately 30% of the total sample flow rate. This percentage can be reduced by slower feed rates occuring over longer periods.

3.0 FEEDSTOCK CONSIDERATIONS

3.1 INTRODUCTION

The success of the bench-scale incineration test program depends upon obtaining samples containing the chemicals to be incinerated in sufficient quantity to provide for the detection of very low concentrations (.01 percent not destroyed by incineration). Such samples must be obtained for liquids, sludges, and soils.

The success of the bench-scale testing program depends on the development of an adequate database concerning the soils, sludges, and liquids to be incinerated. Furthermore, the success of incineration depends upon obtaining sufficient information concerning the feedstocks to ensure safe and complete destruction. Feedstock characterization must be performed with respect to physical, chemical, and thermodynamic properties of the soils, sludges, liquids, and selected major compounds found at Basin F. Of the materials to be incinerated, information exists concerning most of the contaminated chemicals themselves (Table 3.1-1). However, this data set is insufficient to ensure success, and data concerning the soils, sludges, and liquids as a whole are virtually nonexistent.

3.2 SAMPLE CONSIDERATIONS

The principal material to be incinerated is contaminated soil found at Basin F. Soils include both the overburden and the soil beneath the 9.5-mm (3/8-inch) asphalt liner. As a practical matter, the soils to be incinerated will include the asphalt liner as well. Liquid and sludge materials exist in significant quantities, but relative to the soils, are of less consequence.

The hazardous chemicals identified in Table 1.1-1 and 1.1-2 exist in various concentrations in the soils at Basin F. Basin F liquid, however, is considered to be homogeneous. Similarly, the sludges are considered to be relatively homogeneous. The concentrations of chemicals in soils vary as a

TABLE 3.1-1

PROPERTIES OF SELECTED COMPOUNDS

Chemical Compound	Empirical Formula	Molecular Weight	Specific Gravity	State (at 25°C)	Melting Point (C)	Boiling Point (C)	Flash Point (C)	Auto Ignition Temp (C)
1 Chloropane								
1,1 Dichloroethylene	CH_2Cl_2	97		Liquid		31.6	-17.8	570
1,2 Dichloroethylene	ClCHCHCl	97		Liquid	-50	48	2.2	460
2 Chloropane	$CH_3CHClCH_3$	79		Liquid		35.3	-32.2	599.3
Acetophenone	$CH_6COC_6H_5$	120		Liquid	19.7	202.3	82.2	571.1
Aldrin	$C_{12}H_8Cl_6$	365		Solid	104 - 105			
Arsenic	As	75		Solid	814	615		
Benzaldehyde	C_6H_5CHO	106		Liquid	-26	179	64.4	191.7
Benzene	C_6H_6	78	0.880	Liquid	5.5	80.1	-11.1	562.2
Benzoic Acid	C_6H_5COOH	122		Solid	121.7	249	121.1	571.1
Bromo Dichloromethane	$CHBrCl_2$	164		Liquid		89.2 - 90.6		
Carbon Disulfide	CS_2	76		Liquid	-111	46.5	-30	90
Carbon Tetrachloride	CCl_4	154	1.590	Liquid	-22.6	76.8	None	
Chlorobenzene	C_6H_5Cl	113	1.100	Liquid	-45	131.7	29.4	638.3
1-Chlorobutane	$CH_3(CH_2)_2CH_2Cl$	93		Liquid	-123.1	78	-9.4	460
Chloroform	$CHCl_3$	119	1.490	Liquid	-63.5	61.26	None	
Chlorohexane	$C_6H_{13}Cl$	121		Liquid		134	35	
Copper	Cu	64		Solid	1,083	2,324		
Cyclohexane	C_6H_{12}	84		Liquid	6.5	80.7	-20	245
Dieldrin	$C_{12}H_8Cl_6O$	381		Solid	150	175 - 176		
Dihydroxybenzoic acid (methyl ester)	$C_7H_6O_4$	154		Solid	199-200			
Dimethyl Disulfide	$CH_3-S-S-CH_3$	94	1.060	Liquid		109.7 - 115		
Dimethyloxyethane	$CH_3OCH_2CH_2OCH_3$	90		Liquid		83	40	
DIMP (diisopropylmethylphophonate)	$C_7H_{17}PO_3$	193	0.980	Liquid		174		

TABLE 3.1-1 (Continued)

PROPERTIES OF SELECTED COMPOUNDS

Chemical Compound	Empirical Formula	Molecular Weight	Specific Gravity	State (at 25°C)	Melting Point (C)	Boiling Point (C)	Flash Point (C)	Auto Ignition Temp (C)
Diphenylethane	$(C_6H_5)_2CHCH_3$	182		Liquid	-20	272	128.9	440
Dithiane	$C_4H_8S_2$	120		Solid	108 - 113	199 - 200		
DMMP (Dimethylmethylphosphonate)	$C_3H_9PO_3$	124	1.140	Liquid		181		
Endrin	$C_{12}H_8Cl_6O$	381	1.645	Solid	235			
Ethoxyethylene								
Fluoride	F_2	38		Liquid	-218	-187		
Heptane	$CH_3(CH_2)_5CH_3$	100		Liquid		98.5	-3.9	215
Hexachlorobenzene	C_6Cl_6	285			230	326	242.2	
Hexane	$CH_3(CH_2)_4CH_3$	86		Liquid		68.7	-21.7	225
Iron	Fe	56		Solid	1,535	3,000		
Isodrin	$C_{12}H_8Cl_6$	365		Solid	241 - 242			
Magnesium	Mg	24		Solid	651	1,107		
Mercury	Hg	200		Liquid	-38.89	356.9		
Methyl Acetate	$CH_3CO_2CH_3$	74		Liquid	-98.7	57.8	-10	501.7
Methylacetophenone								
Naphthalene	$C_{10}H_8$	128		Solid	80.1	217.9	78.9	526.1
Pentachlorobenzene	C_6HCl_5	250						
Pentachloroethane	$CHCl_2CCl_3$	202		Liquid	-29	162		
Phenol	C_6H_5OH	94		Solid	40.6	181.9	79.4	715
Phosphorus	P	31		Solid	44.1	280	Spont AI	30
Sodium Acetate	$NaC_2H_3O_2$	82		Solid	324			607.2
Sodium Fluoride	NaF							
Sodium Hydroxide	NaOH							
Sodium Methyl Phosphonate								
Sodium Sulfate								
Sodium Sulfate	Na_2SO_4							

TABLE 3.1-1 (Continued)

PROPERTIES OF SELECTED COMPOUNDS

Chemical Compound	Empirical Formula	Molecular Weight	Specific Gravity	State (at 25°C)	Melting Point (C)	Boiling Point (C)	Flash Point (C)	Auto Ignition Temp (C)
Sulfur (Flowers of Sulfur)	S_8	256	2.07	Solid	119	444.6	207	232
Tetrachlorobenzene	$C_6H_2Cl_4$	216	1.73	Liquid	138	245	155	--
Tetrachloroethylene	CCl_2CCl_2	166	1.63	Liquid	-23.4	121.2	None	--
Toluene	$C_6H_5CH_3$	92	0.866	Liquid	-95	110.4	4.4	480
Trichlorobenzene	$C_6H_3Cl_3$	181		Solid	63.4	208.5	107	--
Xylene (Ortho, Meta, and Para)	$C_6H_4(CH_3)_2$	106.2	0.861-0.88	Liquid	-47.9	138.8 - 144.4	27.2 - 32.2	465 - 530

function of borehole location and depth (see Appendix C of the Task 17 Technical Plan). Most concentrations are in the parts per billion (ppb) range, although some concentrations are in the parts per million (ppm) range.

It is not essential that the samples of soil used in the bench-scale incineration testing program contain a representative average of waste concentrations. Average conditions may never be encountered in the actual program. Rather, it is essential that the severe problems be tested explicitly. For this reason, soils from the area of Borehole No. 01 will be used to test the adequacy of the incineration regimes available. The area of Borehole No. 01 has been chosen because it has not lost its asphalt liner. The overburden is particularly contaminated, and the soils beneath the liner also exhibit significant levels of contamination. Borehole No. 01 is located in the area known as "Little F," the area dyked in 1962 and apparently containing the most problematical soils and potential sludges as well as liquids (See Figure 3.1-1).*

3.3 SAMPLE TESTING

Sample testing includes physical, chemical, and thermodynamic properties of the liquids, potential sludges, and soils as well as screening for potential POHCs. Sample testing, therefore, will occur in two phases. All samples to be subjected to the bench-scale incineration system will be homogenized and then characterized for physical, chemical, and thermodynamic (PCT) properties and potential POHCs. Samples will be obtained in 15 kg quantities in order to provide sufficient material for the bench-scale process plus all characterization studies which must precede it.

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*Note: Under Task Order No. 6, Environmental Sciences and Engineering (ESE) is developing the contamination profile of Basin F and soon will send Ebasco a copy of the draft report. This report will define the locations and magnitude of contaminants presently existing in and around Basin F. Upon evaluation of the report, Ebasco may change the soil sampling location.

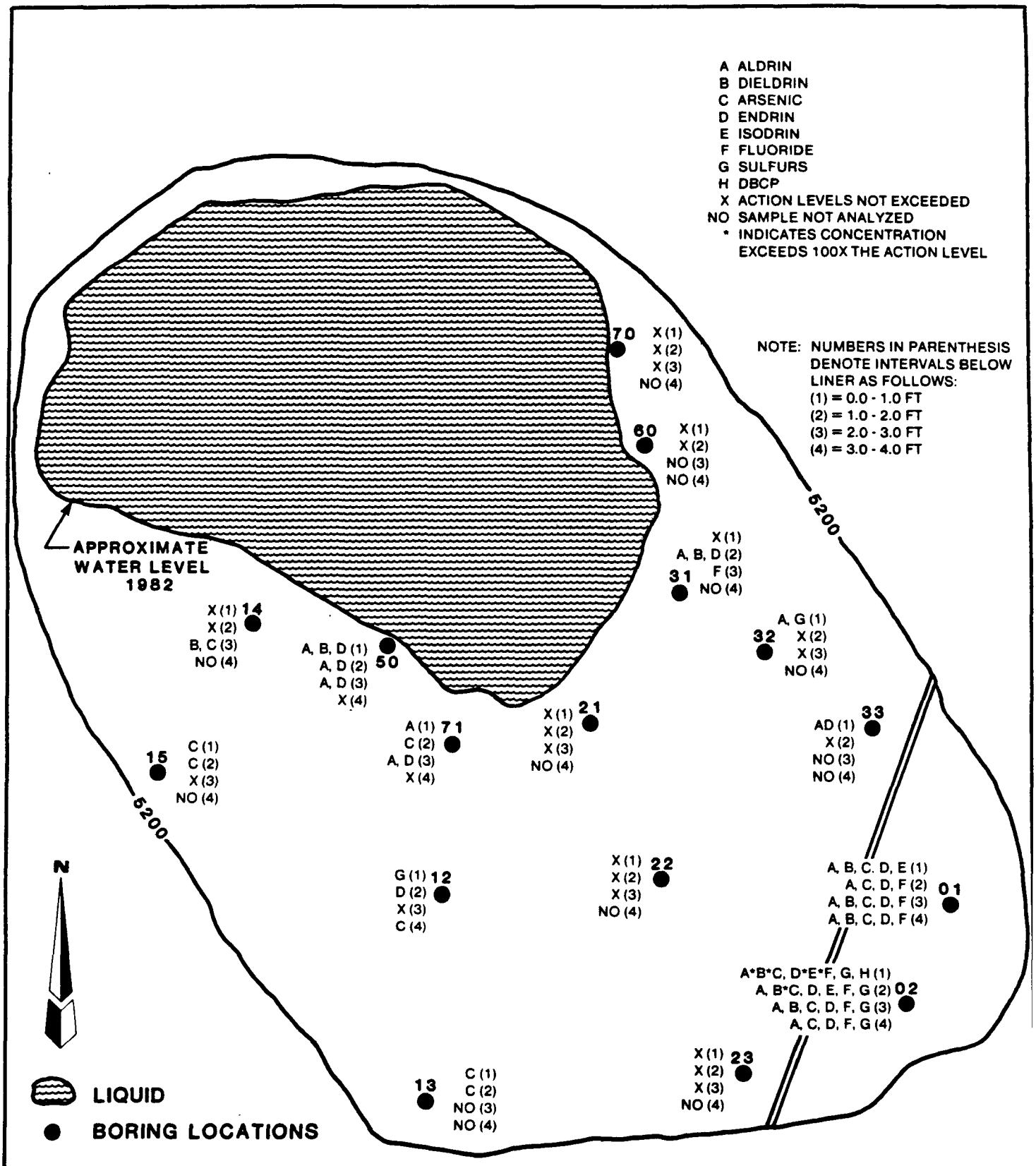


Figure 3.1-1

SOIL SAMPLING LOCATION

SOURCE: MYERS AND THOMPSON, 1982

3.3.1 Feedstock Characterization

The feedstock characterization program is designed to define those PCT properties essential for understanding the bench-scale program and for contributing to the larger-scale operations. Such characterizations are not intended to provide a complete listing of properties, but only such a listing as is essential for safe and cost-effective operation of the system.

Certain physical and chemical properties have already been partially determined for the hazardous chemicals to be destroyed and removed by incineration. Some of these properties include chemical formula, molecular weight, melting point, boiling point, flash point, and autoignition temperature. Heats of combustion either have been determined or calculated, as will be shown in Chapter 4.

Critical parameters are those describing the matrix containing the hazardous chemicals. Those parameters requiring definition are identified below:

Material	Parameters to be Determined
Soils (including overburden and liner)	Specific heat Heat capacity Thermal conductivity Moisture content Ash fusion temperature
Sludges	Viscosity Moisture content Ash fusion temperature Distillation curve
Liquids	Viscosity Corrosivity (with respect to refractory)

These parameters will be determined, to the greatest extent possible, in the evaluation of the samples prior to bench-scale incineration testing.

3.3.2 Analytical Screening for Potential POHCs

Samples collected for test incineration from Basin F will include:

- o Soil;
- o Sludge; and
- o Liquid.

Nonhomogeneity of the collected samples is expected due to the wide variability in soil type and multi-phase characteristics of the liquid/sludge in Basin F. As the lagoon evaporates, the liquid/sludge portion of the basin becomes more concentrated with organic and inorganic constituents. As a result, the concentrations of these compounds can be expected to vary widely across the basin area. Therefore, the samples taken and delivered to the laboratory will not be homogeneous.

An initial screen of the samples may be performed on aliquots of each matrix that has been made as homogeneous as possible by mixing. Enough sample of each matrix type will be collected, homogenized, and stored at the Hittman/Ebasco laboratory at 4°C or less to use for all incineration tests. Chemical characterization of waste samples is critical to evaluating the destruction and removal efficiency (DRE) of incineration tests. The most efficient and cost effective means of characterizing the waste is to:

1. Combine all samples received by matrix into one bulk sample.

2. Homogenize by mixing or agitating the bulk sample.*

* The effectiveness of this process will be evaluated. If found to be unsatisfactory, alternate procedures will be investigated.

3. Prepare aliquots from each bulk matrix for analytical screen.

4. Ship aliquots to appropriate laboratory.

5. Determine the constituent concentration.

Soil, sludge, and liquid samples will be assayed semiquantitatively by gas chromatography/mass spectrometry (GC/MS) for volatile and semivolatile organic target analytes. An attempt will be made to identify other major unknown peaks present in the GC/MS total ion current profiles. Potential unknown analytes will be tentatively identified, if possible. Collected samples will also be assayed quantitatively by graphite furnace atomic absorption spectroscopy for arsenic, by cold vapor atomic absorption spectroscopy for mercury, and for other target metals by inductively coupled argon plasma (ICP) emission spectroscopy. Soils, sludges, and liquids will be characterized in terms of ignitability, corrosivity, and reactivity.

4.0 SELECTION OF TEST PARAMETERS

4.1 INTRODUCTION

The planned bench-scale test consists of 20 test burns, largely on contaminated soils but also including sludges and liquid from Basin F. The 20 test burns are necessary due to the multiple runs required to adequately test for all major contaminants regardless of concentration. Such testing will lead to the ultimate selection of POHCs for pilot-scale and full-scale operations.

A bench-scale test matrix was developed recognizing the typical operating parameters for hazardous waste incinerators capable of handling chemically contaminated solids. These representative parameters are as follows (from Frankel, Sanders, and Vogel 1983).

	Type of Incinerator		
Parameter	Rotary Kiln	Fluid	Hearth
Temperature of Primary Chamber (°C)	280-1,280	750	560-900
Temperature of Afterburner (°C)	900-1,600	N/A	1,000-1,600
Residence Time in Primary Chamber	2 hrs	0.75-2.5 sec	10-30 min
Residence Time in Afterburner	1.3 sec	N/A	2 sec

The parameters shown by Frankel et al. are not the only ones utilized in or reported for incineration. Other authors have shown afterburner residence times of up to 5 seconds for gases evolved in primary incinerators. For example, Bonner et al. (1981) report that

afterburner residence time requirements may be 0.2-6.0 seconds depending upon the waste being destroyed. Bonner also reports varying temperature regimes depending upon technology. The temperatures reported for a fluidized bed in Bonner et al. (1981) are 450-980°C. These are consistent with, but broader than, the temperatures previously mentioned. Dellinger et al. (1984) gives typical afterburner conditions of 2-4 seconds (out of a potential range of 1-12 seconds) and bulk gas temperatures of 600-1,100°C.

The basis for the parameters also includes optimal fuel combustion conditions as discussed in Kramlich et al. (1984), focusing on excess O_2 in the stack gas at 5.4 percent (35 percent excess air), and an upper bound of approximately 7 percent O_2 in the stack (corresponding to about 50 percent excess air). The basis of the parameters includes the limitations of the laboratory equipment, identified as follows:

1. Maximum primary chamber temperature, 800-1,000°C
2. Maximum practical afterburner temperature, 1,250°C

The basis of the test matrix also includes the goals of: 1) obtaining sufficient spread in the parameters to develop first order kinetic approximations of destructive mechanisms (a problem with the residence times of the MRI experiments); and 2) obtaining at least one regime where DRE levels of 99.99 percent are reasonably assured.

4.2 TEST MATRIX PARAMETERS

The test matrix parameters involve varying residence time in the afterburner, temperature in the afterburner, and O_2 concentration in the carrier gas. These parameters are summarized as follows:

	Value	
Parameter	Minimum	Maximum
Time (sec)	2	5
Temp (°C)	900	1,250
O_2 (%)	5.4	7.0

Additional runs will be made at 650°C, as discussed below, in order to ensure that some regimes will be tested that will not succeed. These runs will be at 2 and 5 seconds residence time in the afterburner, but will only be at 5.4% O_2 concentration.

The basis for each parameter is summarized below.

4.2.1 Selection of Time Parameter

The variation in residence time is based upon the values in the literature. The minimum value of 2 seconds appears in virtually all scientific and engineering materials concerning hazardous waste incineration. Dellinger et al. (1984), for example, reports the temperature to achieve destruction of a compound at a DRE of 99.99 percent in 2 seconds (see Table 1.1-3). The residence time of 2 seconds is also consistent with the data presented by Frankel et al. (1983) for afterburners being operated commercially, as shown above.

The maximum value of 5 seconds appears to be a practical upper limit based upon the bench-scale apparatus and the need for firing 300-500 grams of contaminated materials per test to fairly simulate larger-scale operations. The 5-second value appears near the upper end of the scale presented by Bonner et al. (1981). Further, the spread between 2 seconds and 5 seconds provides sufficient range to achieve a fair extrapolation to 6 seconds should such an extrapolation be necessary.

4.2.2 Selection of Temperature Parameter

The laboratory-scale operations will use a primary chamber (Linder furnace) temperature of 1,000°C, or as close to that as can be achieved. It is expected that the actual temperature reached will be between 800 and 1,000°C, rather than the peak value. This temperature is consistent with the values reported by Frankel et al. (1983) for rotary kilns and hearth type furnaces and the values reported by Bonner et al. (1981) for fluidized bed furnaces. The final temperature selected could well be limited by the feedstock tests concerning ash fusion temperatures of the soils to be fed.

The first temperature in the afterburner, 900°C, is consistent with the minimum afterburner temperature reported by Frankel et al. (1984) for afterburners associated with rotary kilns. Further, it is consistent with the literature concerning temperatures required to achieve 99.99 percent destruction (see, for example, Dellinger et al. 1984 as shown in Table 1.1-3). It presents a minimum temperature below which 99.99 percent DRE is probably not achievable.

The maximum temperature in the afterburner, 1,250°C, represents a practical upper limit of the bench-scale equipment. Further, it is in the middle of the range for afterburners as reported by Frankel et al. (1984). Because there is a 350 Centigrade degrees spread between the minimum and maximum temperatures, there is considerable reason for confidence in extrapolating the results to higher temperatures (e.g., 1,500°C) should such extrapolation prove necessary.

A third temperature, 650°C, has been chosen as a minimum value for test purposes. This temperature is consistent with the low end of values shown for afterburners. Further, it is at the low end of temperatures where 99.99 percent DRE for hazardous organics is achieved as shown in Dellinger et al. (1984). The temperature of 650°C is designed to provide a failure to achieve 99.99 percent DRE for some (but not all) compounds in order to provide the most effective data for kinetic calculations. Tests run at 650°C will be at 2 and 5 seconds, but will only be at 5.4 percent O_2 in the carrier gas.

The third temperature provides a matrix of six points for the establishment of time and temperature requirements to incinerate the soils. The matrix appears as follows:

Time	Temperature		
	Minimum	Intermediate	Maximum
2 seconds	650°C	900°C	1,250°C
5 seconds	650°C	900°C	1,250°C

4.2.3 Oxygen Concentration

Oxygen concentration is a parameter chosen to determine the level of excess air which is optimal in firing of the supplementary fuel. Oxygen concentration is varied in the carrier gas as a means of making the bench-scale tests most representative of the postflame oxidation regions as well as the pyrolysis region. Oxygen concentration influences not only the temperatures achieved in the flame (see, for example, Babcock and Wilcox (1978) for a correlation between excess O_2 and flame temperature), but also influences the degree of completeness of combustion and the minimization of PIC formation. The correlation between excess air and excess O_2 in the dry stack gas is shown in the following equation (Babcock and Wilcox 1978):

$$\%EA = 100 \times (O_2 - 0.5\ CO)/(.264N_2 - (O_2 - 0.5\ CO) \quad (1)$$

Where %EA is percent excess air, O_2 is percent oxygen in the dry stack gas, CO is percent carbon monoxide in the dry stack gas, and N_2 is percent nitrogen in the dry stack gas. For these calculations, the CO term can be ignored because proper combustion reduces CO to less than .002-.005% at an absolute maximum (20-50 ppmv CO). Nitrogen concentration can be taken at 79 percent. Consequently, the expression can be simplified to the following:

$$\%EA = 100 \times O_2/(20.856-O_2) \quad (2)$$

Solving this equation for various levels of excess air provides the following values:

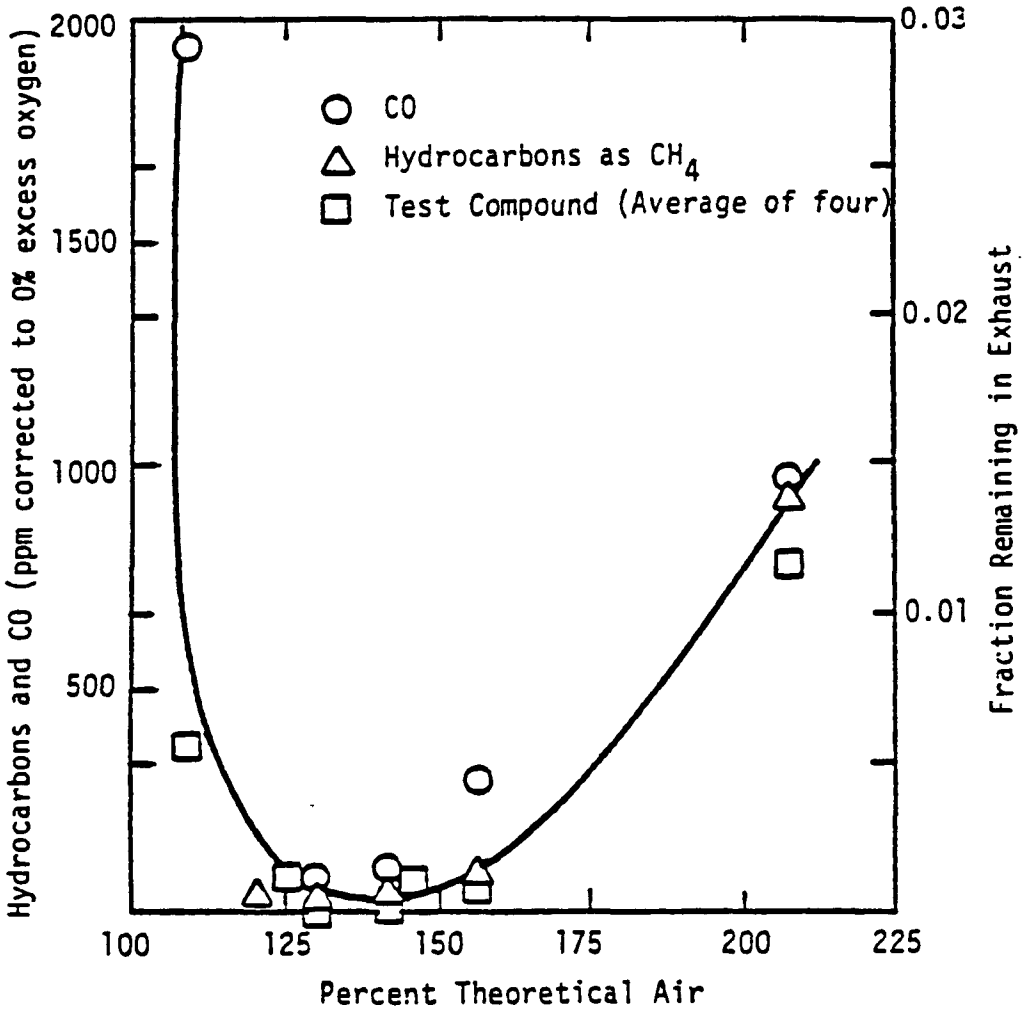
%EA	$\%O_2$
25	4.2
30	4.8
35	5.4
40	6.0
50	7.0
75	8.9

The research by Kramlich et al. (1984) previously cited demonstrates that PICs are minimized and DREs are maximized with excess air in the 30 to 40 percent range. Below and above that range, PICs increase in dramatic quantities, as is shown in Figure 4.2-1.

The minimum concentration of O_2 in the stack gas is selected at 5.4 percent, corresponding to the apparent optimal value shown in Figure 4.2-1. This level can be set for the carrier gas in the experiment. It corresponds to a CO_2 level of 15.6 percent. Further, because 5.4 percent O_2 is an apparent minimum point, selection of any value below this depiction of 35 percent excess air would seriously distort efforts at limited extrapolation. Such distortions would make the results of excess air levels greater than 35-50 percent appear to be more favorable than would be expected under actual operations.

The maximum concentration of O_2 is set at 7.0 percent, corresponding to common firing practices of many combustion systems. Further, this representation of 50 percent excess air represents a practical upper bound beyond which DRE levels of 99.99 percent could not practically be expected (see, for example, Figure 4.2-1). Finally, the spread between 35 and 50 percent excess air does provide sufficient data for limited extrapolation to levels between 50 percent and 75 percent.

It should be noted that the values of 5.4 percent and 7.0 percent represent oxygen concentrations expected for the postflame region. Should it become necessary in order to demonstrate 99.99 percent DRE,



(constant air velocity, variable load, equal molar mixture of chloroform, benzene, chlorobenzene, and acrylonitrile added 3 percent by weight to heptane).

FIGURE 4.2-1

EXHAUST CO AND TOTAL HYDROCARBONS AND FRACTION OF TEST COMPOUND REMAINING IN EXHAUST AS A FUNCTION OF THEORETICAL AIR

one experiment will be run at 5 seconds and 1,250°C with air as the carrier gas in order to more closely approximate flame mode oxygen concentrations.

4.2.4 Test Execution

The regimes established above will be tested on the contaminated soils fractions. Two runs will be made per sample in selected cases in order to ensure adequate data on all hazardous organics identified. Such tests will be run at 1,250°C for 5 seconds at 5.4% O_2, 1,250°C for 2 seconds at 5.4% O_2, 900°C for 5 seconds at 5.4% O_2, and 900°C for 2 seconds at 5.4% O_2. POHCs will be selected for single run tests at 7% O_2 in the carrier gas, and for tests at 650°C. Once a rough optimum regime has been determined for contaminated soils, it will be tested on the liquids (where two runs are contemplated), on sludges (two runs), and on a proportionate mixture of all materials found at Basin F. The two runs on the proportionate mixture of all materials will be at 5 second residence time and at 900°C and 1,250°C temperatures in the afterburner.

4.3 SELECTION OF PRINCIPAL ORGANIC HAZARDOUS COMPOUNDS (POHCs)

POHCs are used as compounds that can measure the fate of all hazardous chemicals to be destroyed. They are chosen based upon thermal stability and concentration. Various ranking schemes commonly proposed for the selection of POHCs include:

- Heat of combustion of the hazardous chemical;
- Autoignition temperature;
- Theoretical kinetics; and
- Thermal decomposition data.

Each methodology has its strengths and weaknesses. Heat of combustion permits evaluation of all compounds either by experimentally determined values in kcal/g, or by calculated values. Heat of combustion, however, does not deal with the issue of thermal stability. Autoignition

temperature, theoretical kinetics, and thermal decomposition data provide additional insights. Unfortunately, the database is incomplete for such properties with respect to the compounds found in Basin F.

The U.S. Environmental Protection Agency (EPA) utilizes heat of combustion for selection of POHCs according to the following formula:

$$\text{POHC rank} = (\%C) + 100/Hc \qquad (3)$$

Where %C represents percentage of concentration in the waste and Hc is heat of combustion in kcal/g. This formula is used here in the absence of more analytically precise kinetics and thermal decomposition data. It is used to recognize that Hc and thermal stability are not necessarily correlated. This formula is more sensitive to Hc than concentration with respect to Basin F wastes due to the low concentrations of materials (typically in the ppb and ppm ranges).

Table 4.3-1 is a compilation of heats of combustion for the hazardous organics in the soils sampled at Boring No. 1 and in the liquids found in Basin F. Of these, aldrin has an Hc of 3.75 kcal/g and endrin, has an Hc of 3.46 kcal/g (Dellinger et al. 1984). These chemicals, along with dieldrin, can be classified as POHCs. Because of the critical nature of these tests, however, and the lack of absolute precision in using the Hc value to determine appropriateness of any POHC with respect to incinerability, the bench-scale tests will be performed initially for all identified compounds in the soils obtained from Basin F Borehole No. 01. This testing for all compounds necessitates multiple (4) runs. Based on these runs, the POHCs will be determined for the remaining tests.

TABLE 4.3-1

HEATS OF COMBUSTION FOR HAZARDOUS WASTES [1/]

Compound	Formula	Btu/lb	Kcal/gram
VOLATILE HALO ORGANICS			
Chloroform (trichloromethane)	$CHCl_3$	1,350	0.75
1,1 - Dichloroethane (ethylidene chloride)	CH_3CHCl_2	5,405	3.00
1,2 - Dichlorethane (ethylene chloride)	$ClCH_2CH_2Cl$	5,405	3.00
1,1,1 - Trichloroethane (methylchloroform)	CH_3CCl_3	3,585	1.99
1,1,2 - Trichloroethane (vinyltrichloride)	Cl_2CHCH_2Cl	3,585	1.99
Tetrachloroethylene (perchloroethylene)	Cl_2CCCl_2	2,145	1.19
Carbon tetrachloride (tetrachloromethane)	CCl_4	430	0.24
1,2 - Trans-dichloroethylene (acetylene dichloride)	ClCHCHCl	4,865	2.70
Dichloromethane (methylene chloride)	CH_2Cl_2	3,065	1.70
Hexachlorobutadiene	C_4Cl_6	3,820	2.12
Hexachloroethane	C_2Cl_6	830	0.46
VOLATILE AROMATICS			
Benzene (benzol)	C_6H_6	18,070	10.03
Toluene (methylbenzene)	$C_6H_5CH_3$	18,270	10.14
Xylene (O-Xylol)	$C_6H_4(CH_3)_2$	18,450	10.24
Ethyl benzene (phenylethane)	$C_6H_5C_2H_5$	18,500 [2/]	10.27

TABLE 4.3-1 (Continued)

HEATS OF COMBUSTION FOR HAZARDOUS WASTES [1]

Compound	Formula	Btu/lb	Kcal/gram
CHLORINATED AROMATICS			
Chlorobenzene (phenyl chloride)	C_6H_5Cl	11,890	6.60
Hexachlorobenzene (perchlorobenzene)	C_6Cl_6	3,225	1.79
1,2,3,4 - Tetrachlorobenzene	$C_6H_2Cl_4$	4,700	2.61
1,2,4 - Trichlorobenzene	$C_6H_3Cl_3$	6,125	3.40
1,2 - Dichlorobenzene	$C_6H_4Cl_2$	8,235	4.57
ORGANOCHLORINE PESTICIDES			
Aldrin [3]	$C_{12}H_8Cl_6$	6,755	3.75
Endrin [4]	$C_{12}H_8Cl_6O$	6,235	3.46
Dieldrin [5]	$C_{12}H_8Cl_6O$	10,200	5.66
Isodrin [6]	$C_{12}H_8Cl_6$	N/A	N/A
Chlordane [7]	$C_{10}H_6Cl_8$	4,880	2.71
Malathion [8]	$C_{10}H_{19}O_6PS_2$	N/A	N/A
Parathion [9]	$C_{10}H_{14}NO_5PS$	6,505	3.61
Azodrin (monocrotophos)	$C_6H_{14}O_5NP$	N/A	N/A
Vapona (DDVP) [10]	$C_4H_7Cl_2O_4P$	N/A	N/A
Hexachlorocyclopentadiene	C_5Cl_6	3,785	2.10
Atrazine [11]	$C_8H_{14}N_5Cl$	N/A	N/A
DDT [12]	$(C_1C_6H_4)_2CHCCl_3$	8,125	4.51
DDE [13]	$C_{14}H_8Cl_4$	9,100	5.05
Oxathiane	N/A	N/A	N/A

TABLE 4.3-1 (Continued)

HEATS OF COMBUSTION FOR HAZARDOUS WASTES 1/

Compound	Formula	Btu/lb	Kcal/gram
Dithiane	N/A	N/A	N/A
Nabam 14/	$C_4H_6N_2NaS_4$		
Maneb 15	$C_4H_6MnN_2S_4$		
Zineb 16/	$C_4H_6MnN_2S_4Zn$		
NONCHLORINATED ALIPHATIC SOLVENTS			
Methylethyl Ketone (butanone)	C_4H_8O	14,538	8.07
Acetone (propanone, dimethyl ketone)	C_3H_6O	13,300 2/	7.38
Methylisobutyl Ketone (hexone)	$(CH_2)_2CHCH_2COCH_3$	N/A	N/A
Dimethyldisulfide (2,3,-dithiabutane)	CH_3-S-S-SH_3	N/A	N/A
OTHERS			
Acetonitrite (methyl cyanide)	CH_3CN	13,280	7.37
Acrylonitrile (vinyle cyanide)	CH_2CHCN	14,285	7.93
Methane	CH_4	23,879 2/	13.25
Pyridine	NCHCHCHCHCH	14,105	7.83
Ethane	C_2H_6	22,320 2/	12.39
Aniline (phenylamine)	$C_6H_5NH_2$	15,730	8.73
Nitrobenzene	$C_6H_5O_2N$	10,810 2/	6.00

1/ All heat contents from determination of the thermal decomposition properties of 20 selected hazardous organic compounds Dellinger et al. 1984.

2/ Chemical Processes. Felder and Rousseau. 1978.

3/ 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4,5,8-dimethanon aphthalene

TABLE 4.3-1 (Continued)

HEATS OF COMBUSTION FOR HAZARDOUS WASTES 1/

4/ 1,2,3,4,10,10-hexachloro-6,7-epoxy,1,4,4a,5,6,7,8,8a-octahydro 1,4,5,8-endo-endo,dimethanonaphthalene)

5/ 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4,5,8-dimethanonaphthalene

6/ 1,2,3,4,10,10-hexachloro-1,4,4a,8,8a hexahydro-1,4,5,8-endo-dimethanonaphthalene

7/ 1,2,4,5,6,7,8,8-octachloro-4,7,methano-3a,4,7,7a-tetrahydroindane

8/ S - (1-2 dicarbethoryethyl) O,O-dimethyldithiophosphate

9/ O,O-dietlyl O-P-nitrophenylphosphorothioate

10/ O,O-dimethyl O-(2,2-dichlorovinyl phosphate)

11/ 2-chloro-4-ethylamino-6-isopropyl amino-s-triazine

12/ 1,1,1-trichloro-2,2-bis(p-chlorophenyl) ethane

13/ 1,1-dichloro-2,2-bis-(p-chlorophenyl)ethylene

14/ Ethylenebis (dithiocarbamic acid) disodium salt

15/ Manganous ethylenebis (dithiocarbonate)

16/ Zinc ethylenebis (dithiocarbamate)

4.4 SELECTION OF ANALYTICAL PARAMETERS

The analyses that will be performed to achieve the objectives of the waste incineration tests were selected to meet the following criteria:

- o Maximize the information;
- o Minimize number of analytical procedures;
- o Utilize current laboratory certification; and
- o Minimize certification efforts.

The analytical program will support four (4) phases of testing:

- o Initial screen of waste feedstock;
- o POHC evaluation tests;
- o Incinerator optimization tests; and
- o Incineration under optimum conditions.

The complete organic analysis will be performed for four (4) test burns which will cover the full range of test conditions to establish the appropriate POHCs. The initial screen of the feedstock wastes has been discussed in Section 3.2.2. Optimization of the incinerator operating conditions requires rapid analytical response to guide subsequent test burns. To achieve a rapid turn-around of the results during the optimization phase, only POHCs will be tested for in the feedstock, the solid residue fraction, and the off-gas. These analyses will not be certified but will be performed using approved methods. However, while certification may not be necessary, some demonstration of the laboratory's ability to detect the required levels will be required.

The 99.99% DRE level in the optimization phase and optimum conditions phase will be determined from the initial feedstock screening analyses. The 99.99% DRE level, will be used to determine the analytical detection limit. The actual DREs will be calculated for the POHC from the analysis of the individual feed sample.

A table of 99.99% DRE levels will be calculated from the initial feedstock screening data for all POHCs that are selected for analysis during the optimum conditions phase. After the optimum conditions for incineration have been established, the following analytical procedures will be performed at HEAI on the feedstock and the incineration by-products:

- o Chlorinated Hydrocarbon Analysis (GC/ECD)
- o Organosulfur Compound Analysis (GC/FPD)
- o Organophosphorous Compound Analysis (GC/NPD)
- o Volatile Organic Analysis (GC/PID and GC/Hall detector)
- o Hydrogen Halides (F^- & Cl^-)
- o Cyanide (Distillation/Colorimetric)
- o Metals (Arsenic by furnace AA, Mercury by cold vapor AA, general metals by ICP)
- o GC/MS Screen

Furthermore, the above procedures will be used to analyze data for feedstock at the time of each optimum conditions burn. Because HEAI has not proposed to certify the above methods, the data resulting from the analysis can provide only a rough estimate of the quantity of analyte present. This will be necessary to establish the best estimate of POHC concentration at time of the burn. The incineration off-gas and residues will be tested to acquire quantitative data to better estimate the destruction and removal efficiency at optimum conditions.

A more detailed discussion of the procedures is described in Section 5.2. The methods were selected based upon the ideal instrument detection limits that each procedure is capable of producing under optimized analytical preparatory conditions.

A GC/MS screen of the organic fraction of the incineration gas sampling train and of the solid residues will be performed on selected test runs. This screen will not continue to be utilized if the desired detection limits cannot be achieved. However, through a GC/MS screen, more data may be aquired about compounds which are detected but not identified by the GC/EPD, NPD, FPD, or Hall detector analyses.

5.0 ANALYTICAL DETAILS

5.1 SAMPLE HANDLING AND SAMPLE FLOW

Hittman/Ebasco Associates Inc. (HEAI) will be the lead laboratory on sample handling and processing. Samples shipped from the field will be homogenized and properly stored under refrigerated conditions by HEAI until analysis or incineration testing. For initial feedstock analysis, the sample will be shipped by overnight express to the approved laboratory. Figure 5.1-1 illustrates the flow of the sample from RMA to the laboratories for analysis.

The soil sample collected will be from a known area of high contamination to facilitate the bulk homogenization and storage of one sample. Sludge and liquid samples should be of a more consistent contaminant concentration range, although "hot" spots can be expected. After aliquoting for feedstock analysis, the samples will then be aliquoted into separate bottles for each test incineration run. The samples will be maintained in tightly sealed glass containers under refrigerated conditions. The lid of each container will be wrapped with Teflon tape and then a layer of parafilm around that to prevent loss of volatiles. However, it can be assumed that some contaminant concentration levels will drop during this period of optimizing the incinerator burns.

The laboratories performing analyses will be CAL, UBTL, and HEAI. HEAI will have the lead on sample preparation and shipment for feedstock analyses. A full set of organic and metal analyses will be performed on each initial soil, sludge, and liquid feedstock sample. HEAI will be responsible for all incinerator test burn sample analysis. The solid and gas fractions collected from the test burns will be analyzed by methods developed at HEAI. Table 5.1-1 summarizes in tabular form, the tests which will be performed and Table 5.1-2 summarizes the number of analyses to be performed.

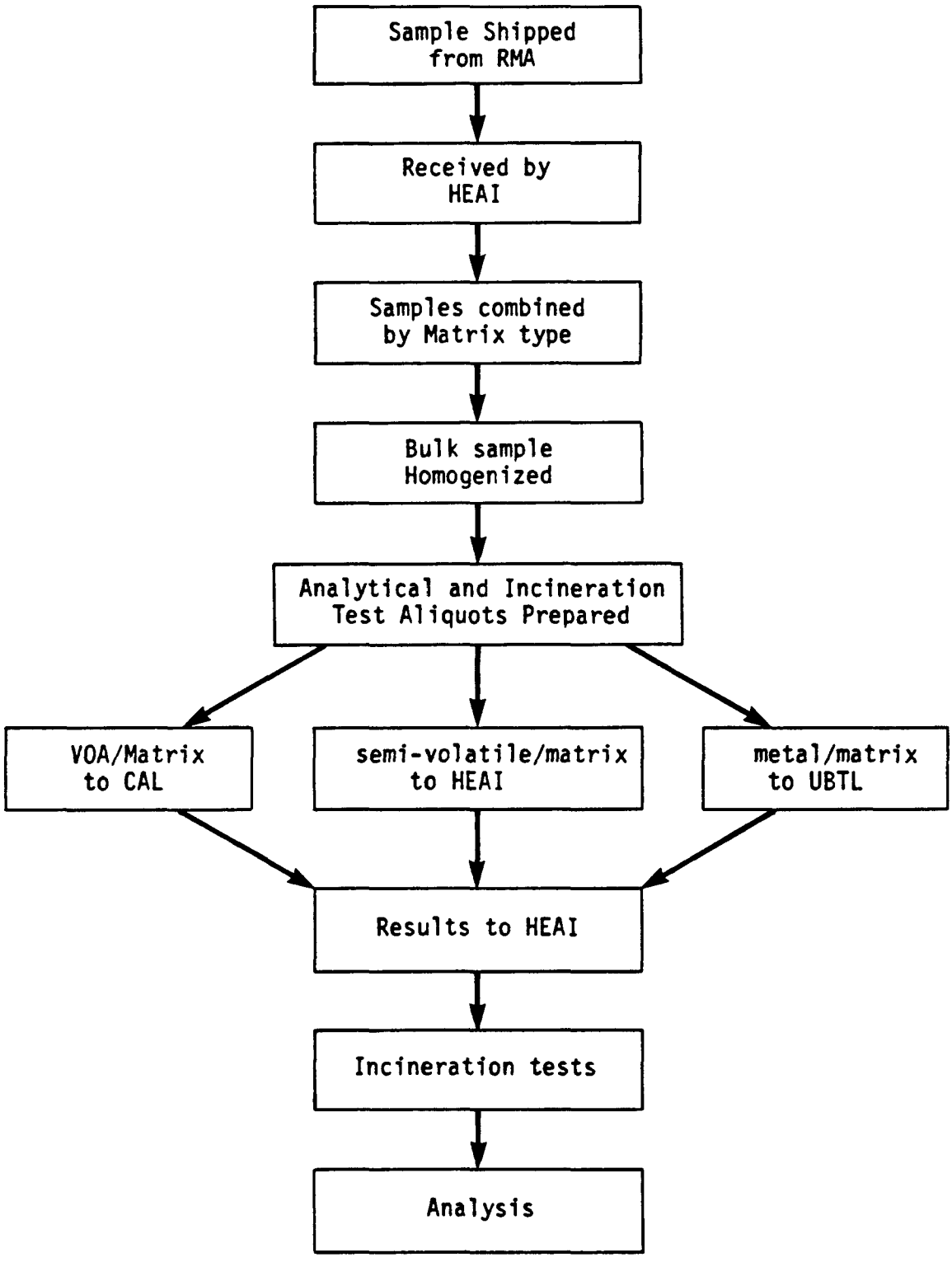


FIGURE 5.1-1
SAMPLE ANALYTICAL FLOW

TABLE 5.1-1

ANALYTICAL METHODOLOGY

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Volatile Organics/Solids			7 days for the solid and 40 days for the extract (1)	Semi-Quantitative (A)	EPA 624 (2) EPA 8240 with EPA 5030 extraction (1) CAL-K9	A 10 gram portion of the sample is obtained with a minimum of handling. The sample is shaken for 4 hours with 10 ml methanol. An aliquot of the methanol extract is injected into 5 ml of water and analyzed by purge-trap GC/MS using a packed column. Surrogates and internal standards are used. Unknowns are identified. Surrogates are: d_2 - Methylene chloride 1,2-Dichloroethane-d_4 d_{10} - Ethylbenzene The internal standard will be 1,2-dibromoethane-d_4.
1,1-Dichloroethane	0.5 µg/g	25 µg/g				
Dichloromethane	0.5 µg/g	25 µg/g				
1,2-Dichloroethane	0.5 µg/g	25 µg/g				
1,1,1-Trichloroethane	0.5 µg/g	25 µg/g				
1,1,2-Trichloroethane	0.5 µg/g	25 µg/g				
Carbon tetrachloride	0.5 µg/g	25 µg/g				
Chloroform	0.5 µg/g	25 µg/g				
Tetrachloroethylene	0.5 µg/g	25 µg/g				
Trichloroethylene	0.5 µg/g	25 µg/g				
Trans-1,2-Dichloroethylene	0.5 µg/g	25 µg/g				
Benzene	0.5 µg/g	25 µg/g				
Toluene	0.5 µg/g	25 µg/g				
Xylene (3 isomers)	0.5 µg/g	25 µg/g				
Ethylbenzene	0.5 µg/g	25 µg/g				
Chlorobenzene	0.5 µg/g	25 µg/g				
Methylisobutyl ketone	0.5 µg/g	25 µg/g				
Dimethyldisulfide	0.5 µg/g	25 µg/g				
Bicycloheptadiene	0.5 µg/g	25 µg/g				
Dicyclopentadiene	0.5 µg/g	25 µg/g				
Semi-Volatile Organics/Solids			7 days for the solid & 40 days for the extract (1)	Semi-Quantitative (A)	EPA 8270 with EPA 3540 extraction (1) HEAI-X9-A	A 15 gram portion of the sample is obtained with a minimum of handling and mixed with 30 grams of anhydrous sodium sulfate. The sample is soxhlet extracted for 8 hours with 300 ml of methylene chloride. The extract is reduced to a final volume of 10 ml in a K-D aparatus. An aliquot of the extract is analyzed by fused silica capillary column GC/MS. Surrogates and internal standards are used. Unknowns are identified.
Aldrin	0.5 µg/g	100 µg/g				
Endrin	0.5 µg/g	100 µg/g				
Dieldrin	0.5 µg/g	100 µg/g				
Isodrin	0.5 µg/g	100 µg/g				
p,p'-DDT	0.5 µg/g	50 µg/g				
p,p'-DDE	0.5 µg/g	100 µg/g				
Chlorophenylmethyl sulfide	0.5 µg/g	100 µg/g				
Chlorophenylmethyl sulfoxide	0.5 µg/g	50 µg/g				
Chlorophenylmethyl sulfone	0.5 µg/g	100 µg/g				

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Hexachlorocyclopentadiene	0.5 µg/g	100 µg/g				Surrogates are:
Oxathiane	0.5 µg/g	100 µg/g				d_4-1,3-Dichlorobenzene
Dithiane	0.5 µg/g	100 µg/g				d_4-Diethylphthalate
Malathion	0.5 µg/g	100 µg/g				d_4-2-Chlorophenol
Parathion	0.5 µg/g	100 µg/g				d_4 Di-n-Octyl Phthalate
Chlordane	0.5 µg/g	100 µg/g				
Azodrin	0.5 µg/g	100 µg/g				The internal standard will be d_{10} Phenanthrene
Vapona	0.5 µg/g	100 µg/g				
Supona	0.5 µg/g	100 µg/g				
DIMP	0.5 µg/g	50 µg/g				
Atrazine	0.5 µg/g	100 µg/g				
ICP Metal Screen/Solids			6 mos (5)	Quantitative (B)	USATHAMA 7S	A 1 gram portion is digested with 3 ml repeated
Cadmium	0.5 µg/g	500 µg/g			UBTL-P9	portions of HNO_3 and finished with HCl. The
Chromium	5 µg/g	500 µg/g				sample is filtered to a final volume of 50 ml.
Copper	5 µg/g	500 µg/g				The sample is analyzed by ICP.
Lead	5 µg/g	500 µg/g				
Zinc	5 µg/g	500 µg/g				
Aluminum	Interelement Correction					
Iron	Interelement Correction					
Arsenic/Solids	1 µg/g	10 µg/g	6 mos	Quantitative (B)	EPA 7060 with EPA 3050 extraction (2) UBTL-B9	A one gram portion of the sample is digested with H_2O_2 + HNO_3. The digest is analyzed by GF/AA.

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Mercury/Solids	0.1 µg/g	1 µg/g	28 days (5)	Quantitative (B)	EPA 245.5 (5) UBTL-Y9	A one gram portion is weighed out and treated with aqua regia followed by potassium permanganate. Excess permanganate is reduced with hydroxylamine sulfate. The mercury is reduced with stannous chloride and determined using the cold vapor technique.
Extraction Procedure Toxicity Incinerator Residues/Solids	-	-	7 days	None	EPA 1310(1) EPA Method C004 (6) HEAI	A 100 gram portion of incinerator residues is extracted for 24 hours with 1.6 liters of deionized water which is maintained at pH 5 ± 0.2 using acetic acid. The extract is analyzed by USATHAMA certified liquid methods shown in Table 6.1-2 for the eight elements, four pesticides and two herbicides listed in 40 CFR 261.24.
Ignitability/Solids	-	-	7 days	None	EPA 1010(1) EPA Method C002 (6) ASTM Method D93-77 HEAI	A sample is heated at a slow constant rate with continual stirring in a cup. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the flame ignites the vapor above the sample.
Corrosivity/Solids	-	-	7 days	None	EPA 1110(1) EPA Method C002(6) NACE Standard TM-10-69 HEAI	Coupons of SAE Type 1020 steel are exposed to the sample and by measuring the degree to which the coupon has been eroded, determines the corrosivity of the sample.

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Reactivity (Total and Amenable Cyanide; and Sulfides)/Solids	-	-	7 days	None	EPA 9010 and EPA 9030(1) EPA Method C003(6) HEAI	Total and Amenable Cyanides: Two 100 gm samples are brought to a 500 ml volume in ASTM type II water. Each sample is then distilled to remove interferences. During distillation cyanide is converted to HCN which is trapped in a scrubber containing 50 ml 1.25 N NaOH. 10-12 drops of rhodamine indicator are added to the scrubber contents. The solution is titrated with standard silver nitrate solution to the first change in color from yellow to brownish pink against an ASTM type II water blank. Sulfides Excess iodine is added to a 50 gm sample which has been treated with zinc acetate to produce zinc sulfide and suspended in 200 ml distilled water. Two ml of 6 N HCl is added to the sample. The iodine oxidizes the sulfide to sulfur under acidic conditions. Excess iodine is back tritrated with sodium thiosulfate using the starch indicator, until the blue color disappears.

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Proximate Analysis: Moisture/Solids	-	-	7 days	None	EPA Method A001a (6) HEAI	A 10 gm soil or 25 gm sludge aliquot is transferred to a tared porcelain evaporating dish. The sample and dish are weighed, then heated on a hot plate to evaporate the sample to near dryness without boiling. The sample and dish are then transferred to a 103°C oven to complete evaporation. Periodically the sample is removed from the oven, cooled in a desiccator and weighed. Dryness is considered complete when weight loss is < 4% of previous weight.
Ash (Loss on Ignition)/Solids	-	-	7 days	None	EPA Method A001b (6) HEAI	After removing a 50 mg aliquot for elemental analysis, the weighed solids from the moisture analysis and porcelain dish are ignited for 30 minutes at 600°C. The ash is cooled in a desiccator and weighed.
Elemental Composition/Solids	-	-	7 days	None	EPA Method A003 (6)	A 50 mg sample of dried solids are analyzed to determine the percent concentration of the following elements: carbon, nitrogen, phosphorus, sulfur, and halogens (i.e. iodine, chlorine, fluorine, bromine).
Heating Value of the Waste/Solids	-	-	7 days	None	EPA Method A006 (6)	A 1 gm sample is placed in a bomb calorimgrer and ignited. The amount of heat released by the burning waste, the activation energy, is expressed as Btu/lb.

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Volatile Halo Organics/Water			14 days (2)	Quantitative	EPA 601 (2)	Purge and Trap GC/Hall Detector with a packed column (1% SP-1000 on Carbopack B) 1,2-dibromo-ethane or other suitable internal standard will be used based on Phase I experience to monitor purge efficiency.[b]
Chlorobenzene	1 µg/L	50 µg/L[a]				
Chloroform	1 µg/L	50 µg/L[a]				
1,1-Dichloroethane	1 µg/L	50 µg/L[a]				
1,2-Dichloroethane	1 µg/L	50 µg/L[a]				
1,1,1-Trichloroethane	1 µg/L	50 µg/L[a]				
1,1,2-Trichloroethane	1 µg/L	50 µg/L[a]				
Tetrachloroethylene	1 µg/L	50 µg/L[a]				
Trichloroethylene	1 µg/L	50 µg/L[a]				
1,2-trans-Dichloroethylene	1 µg/L	50 µg/L[a]				
Dichloromethane	1 µg/L	50 µg/L[a]				
Carbon tetrachloride	1 µg/L	50 µg/L[a]				
Volatile Arom. Organics/Water			7 days (2)	Quantitative	EPA 602 (2)	Purge and Trap/GC/PID with a packed column (1% SP-1000 on Carbopack B, to permit runs in conjunction with EPA 601). A suitable internal standard will be used based on Phase I experience to monitor purge efficiency.[b]
Benzene	1 µg/L	50 µg/L[a]				
Toluene	1 µg/L	50 µg/L[a]				
Xylenes	1 µg/L	50 µg/L[a]				
Ethyl benzene	1 µg/L	50 µg/L[a]				
Organochlorine Pesticides/Water			7 days for the water and 40 days for the extract (2)	Quantitative	EPA 608 (2)	An 800 ml portion of water is extracted with 3 x 50 ml methylene chloride. The extract is reduced in volume and exchanged with iso-octane. The final volume is 10 ml or less. The concentrated extract is analyzed by GC/EC using a fused silica capillary column. Cleanup procedure will be applied as required.[(2)] A suitable internal standard will be selected based on Phase I experience to monitor purge efficiency.[b]
Aldrin	0.1 µg/L	10 µg/L[a]				
Endrin	0.1 µg/L	10 µg/L[a]				
Dieldrin	0.1 µg/L	10 µg/L[a]				
Isodrin	0.1 µg/L	10 µg/L[a]				
Chlordane	0.1 µg/L	10 µg/L[a]				
Hexachlorocyclopentadiene	0.1 µg/L	10 µg/L[a]				
p,p'-DDT	0.1 µg/L	10 µg/L[a]				
p,p'-DDE	0.1 µg/L	10 µg/L[a]				

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Dicyclopentadiene and Bicycloheptadiene/Water	0.3 µg/L	25 µg/L	Extract within 7 days, analyze within 40. See 4 (1)	Quantitative	Developed by MRI for USATHAMA Certification	A 100 ml portion of sample is extracted with 5 ml of methylene chloride. The extract is analyzed by GC/FID using a fused silica capillary column. A suitable internal standard will be specified based on Phase I experience to monitor purge efficiency.
Organosulfur Compounds/Water			Extract within 7 days, analyze within 30. See EPA 625 (1)	Quantitative	USATHAMA 4P	An 800 ml portion is extracted three times with 50 ml methylene chloride. The volume is reduced in a K-D apparatus and exchanged for isooctane. The isooctane extract is analyzed by GC/FPD-S using a packed column (5% SP-1000 on Chromosorb). A suitable internal standard will be specified based on Phase I experience to monitor purge efficiency.
Chlorophenylmethyl sulfide	2 µg/L	50 µg/L				
Chlorophenylmethyl sulfoxide	2 µg/L	50 µg/L				
Chlorophenylmethyl sulfone	2 µg/L	50 µg/L				
1,4 oxathiane	2 µg/L	50 µg/L				
dithiane	2 µg/L	50 µg/L				
Phosphonates/Water			7 days See EPA 625 (1)	Quantitative	USATHAMA 4S for DIMP	An 800 ml portion of the sample is extracted three time with 500 ml methylene chloride. The extract is reduced in volume and exchanged with isoctane. The final volume is 5 ml. The extract is analyzed bya GC/NPD using a fused silica capillary column. Vapona will be added if indicated by Phase I experience. A suitable internal standard will be specified based on Phase I experience to monitor purge efficiency.
Diisopropylmethylphosphonate	2 µg/L	100 µg/L				
Dimethylmethylphosphonate	2 µg/L	100 µg/L			ESE will develop method for DMMP	
Organophosphorous Pesticides/Water			7 days See EPA 625 (1)	Quantitative	EPA 8140(2) modified for water	An 800 ml portion of the samples is extracted three times with 50 ml methylene chloride. The extract is reduced in volume and exchanged with isooctane. The final volume is 5 ml. The extract is analyzed by GC/NPD using a fused silica capillary column. A suitable internal standrd will be specified based on Phase I experience to monitor purge efficiency.
Malathion	0.1 µg/L	5 µg/L				
Parathion	0.1 µg/L	5 µg/L				
Azodrin	0.1 µg/L	5 µg/L				
Supona	0.1 µg/L	5 µg/L				
Vapona	0.1 µg/L	5 µg/L				

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Metals by AA/Water						
Arsenic	10 µg/L	100 µg/L	6 mos (5)	Quantitative	EPA 206.2 (4)	A 100 ml aliquot of sample is digested with H_2O_2 and HNO_3. The digest is analysed by GF/AA.[b]
Mercury	0.1 µg/L	10 µg/L	28 days (5)	Quantitative	EPA 245.1 (4)	A 100 ml aliquot is treated with H_2SO_4, HNO_3, $KMnO_4$, $K_2S_2O_8$. Excess $KMnO_4$ is destroyed with hydroxylamine sulfate. The mercury is reduced with stannous sulfate and analyzed by CV/AA.[b]
Metals by ICP/Water			6 mos (5)	Quantitative	EPA 200.7 (4)	All samples will be treated by adding HNO_3 + HCL and heating before analysis to dissolve precipitates that may have formed after sampling. Magnesium, calcium and sodium may be certified at lower levels if required.[b]
Chromium	50 µg/L	5000 µg/L				
Cadmium	50 µg/L	5000 µg/L				
Lead	50 µg/L	5000 µg/L				
Zinc	50 µg/L	5000 µg/L				
Copper	50 µg/L	5000 µg/L				
Magnesium	10 mg/L	1000 µg/L				
Calcium	100 mg/L	1000 µg/L				
Sodium	100 mg/L	1000 µg/L				
Ignitability/Water	-	-	7 days	None	EPA 1010 (2) ASTM Method D93-77 and EPA Method C001(6)	A liquid sample is heated at a slow constant rate with continual stirring in a cup. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the flame ignites the vapor above the sample.[b]
Corrosivity/Water	-	-	7 days	None	EPA 1110 (2) NACE Standard TM-01-69 and EPA Method C002 (6)	Coupons of SAE Type 1020 steel are exposed to the sample and by measuring the degree to which the coupon has been eroded, determines the corrosivity of the sample.[b]

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Reactivity (Total and Amenable Cyanide, and Sulfide)/Water	-	-	7 days	None	EPA 9010 EPA 9030 (2): and EPA Method C003 (6)	Total and Amenable Cyanides: Two 500 ml samples preserved with 2 ml 1N NaOH are prepared. One is chlorinated to destroy succeptable complexes. Each sample is then distilled to remove interferences. During distillation, cyanide is converted to HCN which is trapped in a scrubber containing 50 ml 1.25N NaOH. Ten to twelve drops of rhodamine indicator are added to the scrubber contents. This solution is titrated with standard silver nitrate solution to the first change in color from yellow to brownish pink against an ASTM Type II water blank. Sulfides: Excess iodine is added to a 200 ml sample which is treated with zinc acetate to produce zinc sulfide. Two ml of 6N HCl is added to the liquid. The iodine, oxidizes the sulfide to sulfur under acidic conditions. Excess iodine is back titrated with sodium thiosulfate, using the starch indicator, until the blue color disappears.[b]
Proximate Analysis: Moisture/Water	-	-	7 Days	None	EPA Method A001a(6)	A 100 ml liquid aliquot is transferred to a tared procelain evaporating dish. The sample and dish are weighed, then heated on a hot plate to evaporate the sample to near dryness without boiling . The sample and dish are then transferred to a 103°C oven to complete evaporation. Periodically the sample is removed from the oven, cooled in a desiccator and weighed. Dryness is considered complete when weight loss is < 4% of previous weight.[b]

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Ash (Loss on Ignition)	-	-	7 days	None	EPA Method A001b(6)	After removing a 50 mg aliquot for elemental analysis, the weighed solids from the moisture analysis and procelain dish are ignited for 30 minutes at 600°C. The ash is cooled in a desiccator and weighed.[b]
Elemental Composition/Water	-	-	7 days	None	EPA Method A003(6)	A 50 ml sample is analyzed to determine the percent concentrations of the following elements: carbon, nitrogen, phosphorus, sulfur and halogens (i.e. iodine, chlorine, fluorine, bromine).[b]
Heating Value of the Waste/Water	-	-	7 days	None	EPA Method A006(6)	A 1 gm sample is placed in a bomb calorimeter and ignited. The amount of heat released by the burning waste, the activation energy is expressed as Btu/lb.[b]
Viscosity/Water	-	-	7 days	None	EPA Method A005(6)	Viscosity is determined by measuring the time in seconds it takes for a fixed volume of liquid sample to flow through the capillary of a calibrated viscometer.[b]
Particulate Mass Loading/Off-Gas	-	-	7 days	None		Glass fiber filter is oven dried and cooled in a dessicator to a constant weight with a minimum amount of handling.[b]

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Volatile Organics/Off-Gas	--	--	4 weeks in freezer	None		The front and back sections of the Tenax tubes are combined and thermally desorbed. The desorbed organics are analyzed by GC Hall using a fused silica capillary column.[b]
Acid Gases/Off-Gas	--	--	28 days	None		The 0.1n NaOH sorbent from the stack gas impinger is assayed by specific ion probe for chloride.
Volatile Metals/Off-Gas			6 mos	None	EPA 200.7(5)	An aliquot of silver catalyzed ammonium persulfate sorbent is treated with HNO_3 + HCl and heated before analysis to dissolve precipitates and analyzed by ICP.[b]
Chromium	50 µg	500 µg				
Cadmium	50 µg	500 µg				
Lead	50 µg	500 µg				
Zinc	50 µg	500 µg				
Copper	50 µg	500 µg				
Volatile Metals/Off-Gas						
Arsenic	1 µg	10 µg	6 mos	None	EPA 206.2 (5)	An aliquot of silver catalyzed ammonium persulfate sorbent is treated with H_2O_2 and HNO_3. The digest is assayed by GF/AA.[b]
Volatile Metals/Off-Gas						An aliquot of silver catalyzed ammonium persulfate sorbent is treated with H_2SO_4, HNO_3, $KMNO_4$ and $K_2S_2O_8$. Excess $KMNO_4$ is destroyed with hydroxylamine sulfate. The mercury is reduced with stannous sulfate and analyzed by cold vapor AA.[b]
Mercury	0.1 µg	10 µg	28 days	None	EPA 245.1 (5)	

TABLE 5.1-1 (Continued)

Analysis/Matrix/Analytes	Detection Limit*	High Range Concentration	Hold Time	Level of Certification	Reference Methods	Principle of Method
Organophosphorous Compounds/ Off-Gas				Qualitative	Developed by HEAI	XAD-2 sorbent, incinerator residues, fly ash, and water extracted (if necessary). All extracts combined to one sample concentrate. Solvent exchanged as necessary to perform instrumental analysis. Analyses by GC with specific detectors as described under liquid matrix.
Organosulfur Compounds/Off-Gas				Qualitative	Developed by HEAI	
Organochlorine Compounds/Off-Gas				Qualitative	Developed by HEAI	

*Actual detection limits for certified methods are identified in Volume IV of the RMA Procedures Manual (Project Specific Analytical Methods Manual) for each laboratory. Detection limits for uncertified methods and methods to be certified are desired detection limits.

[a] Reflects an estimate of the linear range of the method and is proposed to minimize dilutions.
[b] To be developed during USATHAMA Phase II certification.

References:

(1) EPA SW-846, 2nd ed., "Test Methods for Evaluating Solid Waste".
(2) EPA-600/4-82-057, July 1982 "Methods for Organic Chemical Analysis of Principal and Industrial Wastewater".
(3) Personal Communication from Chris Weathington, Ebasco QA Manager.
(4) ESE-AMP.2-UD-$H_2$0.1, July 22, 1982.
(5) EPA-600/4-79-020, Revised March 1983, "Methods for Chemical Analysis of Water and Wastes".
(6) EPA-600/8-84-002, February 1984, Sampling and Analysis Methods for Hazardous Waste Combustion.

Notes:

(A) Semi-Quantitative: See Section III of the Litigation Technical Support and Services Rocky Mountain Arsenal Procedures Manual, Seciton 11.2.2.1.
(B) Quantitative: See Section III of the Litigation Technical Support and Services Rocky Mountain Arsenal Procedures Manual, Section 11.2.2.1.

Table 5.1-2

Number of Analyses

Test Phase	Analysis	Feedstock			Residual Ash	Off-Gas
		Solid	Liquid	Sludge		
Initial Screen of Waste Feedstock	VOA-GC/MS[1]	1	1	1	-	-
	Semivol.-GC/MS[2]	1	1	1	-	-
	Metals[3]	1	1	1	-	-
POHC Evaluation Tests	VOA-GC/MS	4	-	-	-	-
	Semivol.-GC/MS	4	-	-	-	-
	GC/ECD[4]	-	-	-	4	4
	GC/FPD[5]	-	-	-	4	4
	GC/NPD[6]	-	-	-	4	4
	GC/PID[7]	-	-	-	4	4
Optimization Tests	GC/ECD	12	-	-	12	12
	GC/?[8]	12	-	-	12	12
Test of Optimum Conditions	VOA-GC/MS	1	1	1	-	-
	Semivol.-GC/MS	1	1	1	-	-
	GC/ECD	-	-	-	3	3
	GC/FPD	-	-	-	3	3
	GC/NPD	-	-	-	3	3
	GC/PID	-	-	-	3	3
	Metals	1	1	1	3	3

1. VOA-GC/MS: Volatile Organic Analyses by Gas Chromatography/Mass Spectrometer.

2. Semivol. - GC/MS: Semivolatile Organic Analyses by Gas Chromatography/Mass Spectrometer

3. Metals: Selected metals by inductively coupled plasma and atomic absorption.

4. GC/ECD: Chlorinated Hydrocarbons by Gas Chromatography with Electron Capture Detector.

5. GC/FPD: Organosulfur Compounds by Gas Chromatography, with Flame Photometric Detector.

6. GC/NPD: OrganoPhosphorous Compounds by Gas Chromatography with Nitrogen Phosphorous Detector.

7. GC/PID: Volatile Organic Analysis by Gas Chromatography with Photoionization Detector.

8. Detector depends on selected POHCs.

5.2 ANALYTICAL PROTOCOL SUMMARY

The following is a summary of the analytical procedures which will be followed to support the Task 17 objectives. All soil, sludge, sediment, incineration residue, and solid matrices were considered as soils for analytical purposes. Analytical methods, target analytes, and desired target detection limits for liquid matrix analytes are discussed in this section as well.

The off-gas analytical procedures have not been developed in detail but a summary of the analytical approaches and procedures that may be expected to meet the requirement of Task 17 are listed.

5.2.1 Volatile Organics in Soil and Solid Samples by Gas Chromatography/Mass Spectrometry (GC/MS)

The volatile organics method for solids was based on EPA Method 8240 (EPA SW-846). This method was PMO certified for soils and solids at the semiquantitative level for the Task 17 Program (USATHAMA Method N9 for UBTL and K9 for CAL).

In this method, a 10-gram portion of the sample will be obtained with minimum of handling and placed into 10 ml of methanol in a volatile organic acid (VOA) septum vial, spiked with the surrogates: methylene chloride-d_2; 1,2 Dichloroethane-d_4; and ethyl benzene-d_{10}, capped with a teflon lined lid, and shaken for four hours. A 20-ug aliquot of the methanol extract will be removed, spiked with 200 ug of 1,2-dibromoethane-d_4 as an internal standard, and injected into 5 ml of organics-free water and contained in a syringe. The contents of the syringe will then be injected into a purging device, purged, and analyzed on a packed column (1% SP-1000 on Carbopack B) by GC/MS. Each sample will be assayed for target compounds at detection limits identified in Table 5.1-1.

In addition, the total ion current profile will be screened for all major unknown peaks. An attempt will be made to identify the largest of these major unknown peaks which are present in excess of ten percent of the area of the internal standard peak. Each of these major unknown peaks will be reported as the purity, fit and probability to match the three most likely candidate compounds from the Environmental Protection Agency/National Bureau of Standards/National Institute of Health (EPA/NBS/NIH) Mass Spectral library computer program.

5.2.2 Semivolatile Organics in Soil and Solid Samples by Gas Chromotography/Mass Spectrometry (GC/MS)

This analytical technique was based on EPA Method 8270 for solids (EPA SW-846) and was PMO certified for soils and solids at the semiquantitative level for the Task 17 program (USATHAMA Method L9 for UBTL, X9 for CAL, and X9-A for HEAI).

Using this method, a 15-gram portion of the sample will be obtained with a minimum of handling and spiked with the surrogates: 2-chlorophenol-d_4, 1,3-dichloro-benzene-d_4, diethyl phthalate-d_4, and di-n-octyl phtalate-d_4. The sample will be mixed with anhydrous sodium sulfate (30 grams or more depending on sample moisture content) then the soxhelet extracted for 8 hours with 300 ml methylene chloride. The extract is reduced to a final volume of 10 ml in a Kuderna-Danish (K-D) apparatus. An aliquot of this concentrate will be spiked with phenanthrene-d_{10} as an internal standard and analysed on a fused silica capillary column by GC/MS. Samples will be assayed for target analytes at the detection limits shown in Table 5.1-1. In addition, the total ion current profile will be scanned for major unknown peaks. As discussed for volatile organics, an attempt will be made to identify these unknown major peaks.

5.2.3 Metals in Soil and Solid Samples by Inductively Coupled Argon Plasma (ICP) Emission Spectrometry

The ICP method, based on USATHAMA Method 7S, is PMO certified at the quantitative level (USATHAMA Method P9 for UBTL and A9 for CAL).

In this procedure, a one-gram portion of sample will be digested in a watch glass covered Griffin beaker with 3 ml of concentrated nitric acid. Contents of the beaker will be heated to near dryness and repeated portions of concentrated nitric acid will be added until the sample is completely digested. The digestion process is finished with two ml of 1:1 nitric acid and 2 ml of 1:1 hydrochloric acid. The sample digest will be filtered, the beaker and watch glass rinsed with deionized water, and the rinsate passed through the filter. The digestate is brought to a final volume of 50 ml and assayed by ICP.

The sample will be assayed for target metals at detection limits identified in Table 5.1-1.

5.2.4 Arsenic in Soil and Solid Samples by Graphite Furnace Atomic Absorption (AA) Spectroscopy

The arsenic method for soils and solids was developed from EPA Method 7060 (EPA-SE-846). Using this method, a one-gram sample will be digested with hydrogen peroxide and concentrated nitric acid. The digest will be filtered and assayed by graphite furnace atomic absorption spectroscopy. The target detection limit for arsenic will be 1 ug/g. This method is PMO certified at the quantitative level (USATHAMA Method B9 for UBTL and G9 for CAL).

5.2.5 Mercury in Soil and Solid Samples by Cold Vapor Atomic Absorption (CVAA) Spectroscopy

This mercury method, developed from EPA Method 245.5 (EPA 600/4-82-057), is PMO certified at the quantitative level (USATHAMA Method Y9 for UBTL and J9 for CAL). In the method, a one-gram sample portion will be digested with aqua regia followed by treatment with potassium permanganate. Excess permanganate will be reduced with hydroxylamine sulfate. Mercury will be reduced with stannous chloride and assayed by CVAA. The target detection limit for mercury will be 0.1 ug/g.

5.2.6 Extraction Procedure (EP) Toxicity Protocol for Soils, Incineration Residues, and Solids

This extraction procedure is based upon EPA Method 1310 (EPA-SW-846) and EPA Method C004 (EPA-600/8-84-002). The procedure will not be PMO certified. In the extraction procedure, a 100-gram portion of the sample is extracted for a period of 24 hours with 1.6 l of deionized water which is maintained at pH 5.0 $\pm$ 0.2 using 0.5 N acetic acid and monitored throughout the course of the extraction. The sample slurry is allowed to stand to permit the solid phase to settle and the liquid portion to be decanted for filtration. The filtered liquid is the extract. This liquid will be assayed using PMO certified methods for arsenic, cadmium, chromium, endrin, lead, and mercury, and approved methods for selenium, silver, barium, lindane, methoxychlor, toxaphene, 2,4-dichlorophenoxy acetic acid, and 2,4,5-trichlorophenoxy propionic acid.

5.2.7 Ignitability in Soil and Solid Samples

This method is based on EPA Method 1010 (SW-846). Ignitability is determined by heating a sample at a slow, constant rate with continual stirring in a Pensky-Martin closed-cup tester. A small flame is directed into the cup at regular intervals with a simultaneous interruption at which the test flame ignites the vapor above the sample. This method will not be PMO certified.

5.2.8 Corrosivity Toward Steel in Soil and Solid Samples

The corrosivity method is based on EPA Method 1110 (SW-846). In the method, coupons of SAE Type 1020 steel are exposed to the waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, the corrosivity of the waste is determined. This method will not be PMO certified.

5.2.9 Reactivity in Soils and Solid Samples

Reactivity for soils and solids in this task is defined in terms of cyanide or sulfate concentrations. The assay employs EPA Method 9010 (EPA-SW-846) for total and amenable cyanide and EPA method 9030 (EPA-SW-846) for sulfide. For cyanide, a sample will be split into two, 100-gram aliquots, each brought to a 500 ml volume with ASTM Type II water in a 1-liter boiling flask. One aliquot is chlorinated with calcium hypochlorite to destroy susceptable complexes. Each aliquot is distilled to remove interferences and 25 ml of concentrated sulfuric acid is slowly added to each flask. During distillation, cyanide is converted to HCN which is then trapped in a scrubber containing 50 ml 1.25 N NaOH. Ten to twelve drops of rhodamine indicator are added to the scrubber contents. This solution is titrated with standard silver nitrate solution to the first change in color from yellow to brownish pink against an ASTM Type II water blank.

Sulfides are determined by adding excess iodine to a 50-gram sample suspended in 200 ml distilled water which has been treated with zinc acetate to produce zinc sulfide. Two ml of 6N hydrochloric acid is added to the sample. The iodine oxidizes the sulfide to elemental sulfur under acidic conditions. Excess iodine is back titrated with sodium thiosulfate using starch indicator until the blue color disappears. These methods will not be PMO certified.

5.2.10 Proximate Analysis of Soils and Solid Samples

The proximate analysis provides data relating to the physical form of the sample and provides an approximate mass balance as to its composition. This analysis is based upon EPA Method A001a for particulate and moisture, EPA Method A001b for ash (loss on ignition), EPA Method A003 for elemental composition, and EPA Method A006 for the heating value of the sample (EPA-600/8-84-002). Proximate analyses procedures will not be PMO certified.

In the particulate and moisture method (EPA A001a), 10 grams of soil and 25 grams of sludge are placed in a tared porcelain evaporation

dish. The sample and the dish are weighed, then heated on a hot plate to evaporate the sample to the near dryness without scorching. The sample and dish are then transferred to a 103°C oven to complete evaporation. Periodically, the sample and dish are removed from the oven, cooled in a desiccator and weighed. Dryness is considered complete when weight loss is less than 4% of the previous weight.

Ash (loss on ignition) content (EPA A001b) is determined on the weighed solids from the moisture analysis. After removing a 50-mg aliquot for elemental analysis, the solids and procelain dish are ignited for 30 minutes at 600°C. The resultant ash and porcelain dish are cooled in a desiccator and weighed.

The elemental composition method (EPA A003) uses 50 mg of dried solids to determine the percent concentrations of carbon, nitrogen, phosphorous, sulfur, and halogens (iodine, chlorine, fluorine, and bromine). Carbon is determined by measuring carbon dioxide and water upon combustion (ASTMD-3178-73). Nitrogen is determined by the Kjeldahl digestion method (ASTM D-3179-73), and oxygen by the difference method (ASTMD-3176-74). Phosphorous is determined by the spectroscopic method (ASTMD-2795), sulfur by sulfate titration (ASTMD-3177), and halides by halide titration (ASTMD-2361-66).

Heating value of the sample will be determined using the ASTMD-2015 method. In the method, a one-gram sample is placed in a calibrated isothermal jacket bomb calorimeter under controlled conditions. Calorific values (Btu) will be computed from temperature observations made before, during, and after combustion of the sample.

5.2.11 Unknown Identification in Soil, Solid, and Sludge Samples by Gas Chromatography/Mass Spectrometry (GC/MS)

The total ion current profile will be screened for all major unknown peaks. The laboratories will report (RT Code, estimated concentrations and print MS traces) all unknowns with peaks greater than 10 percent of the internal standard response. Each of these major unknown peaks greater than 10 percent of the internal standard response (excluding obviously meaningless peaks, e.g., column bleeds) will be reported as

the purity, fit, and probability to match the three most likely candidate compounds from the Environmental Protection Agency/National Bureau of Standards/National Institute of Health (EPA/NBS/NIH) Mass Spectral library computer program.

5.2.12 Volatile Halogenated Organics in Liquid Samples

The analytical method for volatile halogenated organics in water will be based on EPA Method 601 (EPA 600/4-82-057). This analytical procedure will be a purge and trap method, assayed on a packed column (1% SP-1000 on Carbopack B) by GC equipped with a Hall electrolytic conductivity detector. Water samples will be spiked with 1,2-dibromethane, or another suitable internal standard based on project experience, to monitor purge efficiency.

Volatile halogenated organic analyses and desired detection limits are identified in Table 5.1-1.

5.2.13 Volatile Aromatic Organics in Liquid Samples

The volatile aromatic hydrocarbon methods will be based on EPA Method 602 (EPA 660/4-82-057) for water and EPA Method 8020 (EPA-SW-846) for soil and solids. Analysis of volatile aromatics in water will be a purge and trap method, analyzed by GC equipped with a photoionization detector using a packed column (1% SP-1000 on Carbopack B).

Table 5.1-1 lists the volatile aromatic organic constituents and target detection limits.

5.2.14 Organochlorine Pesticides in Liquid Samples

The analytical methodology for organochlorine pesticides will be based on EPA Method 608 (EPA 600/4-82-057) and EPA Method 8080 (EPA 600/4-82-057) for water and EPA Method 8080 (EPA-SW-846) for soil and solid samples. An 800-ml portion of water will be extracted three times with 50 ml of methylene chloride. The extract shall be reduced

in volume and exchanged with hexane to a final volume of 10 ml or less. The concentrated extract will be analyzed by GC with an electron capture detector using a fused silica capillary column.

Organochlorine pesticides and their target detection limts are listed in Table 5.1-1.

5.2.15 Organosulfur Compounds in Liquid Samples

The organosulfur compounds that will be target analytes are listed in Table 5.1-1. Methodologies for organosulfur analyses will be developed from USATHAMA Method 4P for water. In a water matrix, an 800-ml sample will be extracted three times with 50 ml of methylene chloride. The extract volume shall be reduced in a K-D apparatus and exchanged with isooctane. The isooctane extract will be assayed on a packed column (5% SP-1000 on Chromosorb) by GC with a flame photometric detector. The target detection limit for organosulfur compounds in water will be 2 ug/l.

5.2.16 Organophosphorous Pesticides in Liquid Samples

Organophosphorous compounds targeted for analysis are listed in Table 5.1-1. Analytical methods for these compounds are derived from EPA Method 8140 (EPA-SW-846) for water.

In a water matrix, the five organophosphorous compounds will be extracted from an 800-ml sample with three 50-ml volumes of methylene chloride. The extract will be concentrated and exchanged with isooctane to a final volume of 5 ml. An aliquot of the extract will be assayed on a packed column 6 feet by 2mm ID 1.5% OV17 + 1.95% QF-1 by GC equipped with a nitrogen/phosphorous detector. Target detection limits for the five organophosphorous pesticides in water will be 0.1 ug/l.

5.2.17 Phosphonates in Liquid Samples

The phosphonates include diisopropylmethylphosphonate (DIMP) and dimethylmethylphosphonate (DMMP). Specific analytical methodologies for phosphonates will be developed from USATHAMA Method 4S for water.

The sample will be analyzed on a fused silica capillary column by GC equipped with a nitrogen/phosphorous detector. The target detection limit for phosphonates in water will be 2 ug/l.

5.2.18 Metals in Liquid Samples

Ten metals will be assayed in liquid matrices. The metals and principal analytical method will be as follows: arsenic and mercury by atomic absorption; and cadmium, calcium, chromium, copper, lead, magnesium, sodium, and zinc by ICP.

The method for arsenic analysis will be derived from EPA Method 206.2 (EPA 600/4-79-020) for water. Using EPA Method 206.2 (EPA 600/4-79-020), a 100-ml sample of water will be digested with hydrogen peroxide and concentrated nitric acid. The digest will be assayed by graphite furnace atomic absorption spectroscopy. Target detection limits for arsenic in water will be 10 ug/l.

The mercury methods will be derived from EPA Method 245.1 (EPA 600/4-79-020) for water. In the water method, a 100-ml sample will be treated with sulfuric acid, nitric acid, potassium permanganate, and potassium pursulfate. Excess permanganate will be destroyed with hydroxylamine sulfate. Mercury will be reduced with stannous sulfate and assayed by cold vapor atomic absorption spectroscopy. The target detection limit for mercury in water will be 0.1 ug/l.

The method for ICP metals in water was derived from EPA Method 200.7 (EPA 600/4-79-020). Target analytes and desired detection limits for ICP metals in the liquid matrix is shown in Table 5.1-1.

All water samples for ICP metals will be digested by adding nitric and hydrochloric acid and heating before analyses to dissolve any precipitates that may have formed after sampling. The sample digest will be filtered, brought to a final volume of 50 ml, and assayed by inductively coupled argon plasma emission spectrometry.

5.2.19 Ignitability in Liquid Samples

This method is based on EPA Method 1010 (SW-846). Ignitability is determined by heating a sample at a slow, constant rate with continual stirring in a Pensky-Martin closed-cup tester. A small flame is directed into the cup at regular intervals with a simultaneous interruption of stirring. The flash point is defined as the lowest temperature at which the test flame ignites the vapor above the sample. This method will not be PMO certified.

5.2.20 Corrosivity Toward Steel in Liquid Samples

The corrosivity method is based on EPA Method 1110 (SW-846). In the method, coupons of SAE Type 1020 steel are exposed to the waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, the corrosivity of the waste is determined. This method will not be PMO certified.

5.2.21 Reactivity in Soils and Solid Samples

Reactivity in soils and solids for this task is defined in terms of cyanide or sulfide concentrations. The assay employs EPA Method 9010 (EPA-SW-846) for total and amenable cyanide and EPA Method 9030 (EPA-SW-846) for sulfide. For cyanide, a sample will be split into two, 500-ml aliquots in a 1-liter boiling flask. One aliquot is chlorinated with calcium hypochlorite to destroy susceptable complexes. Each aliquot is distilled to remove interferences and 25 ml of concentrated sulfuric acid is slowly added to each flank. During distillation, cyanide is converted to HCN which is trapped in a scrubber containing 50 ml 1.25 N NaOH. Ten to twelve drops of

rhodamine indicator are added to the scrubber contents. This solutions is titrated with standard silver nitrate solution to the first change in color from yellow to brownish pink against an ASTM Type II water flask.

Sulfides are determined by adding excess iodine to a 200-ml sample which has been treated with zinc acetate to produce zinc sulfide. Two ml of 6N hydrochloric acid is added to the sample. The iodine oxidizes the sulfide to elemental sulfur under acidic conditions. Excess iodine is back titrated with sodium thiosulfate using starch indicator until the blue color disappears. These methods will not be PMO certified.

5.2.22 Proximate Analysis of Liquid Samples

The proximate analysis provides data relating to the physical form of the sample and provides an approximate mass balance as to its composition. This analysis is based upon EPA Method A001a for moisture, EPA Method A001b for ash (loss on ignition), EPA Method A003 for elemental composition, and EPA Method A006 for the heating value of the sample (EPA-600/8-84-002). Proximate analyses procedures will not be PMO certified.

In the moisture method (EPA A001a), a 100-ml liquid sample is placed in a tared porcelain evaporation dish. The sample and the dish are weighed, then heated on a hot plate to evaporate the sample to near dryness without scorching. The sample and dish are then transferred to a 103°C oven to complete evaporation. Periodically, the sample and dish are removed from the oven, cooled in a desiccator, and weighed. Dryness is considered complete when weight loss is less than 4% of the previous weight.

Ash (loss on ignition) content (EPA A001b) is determined on the weighed solids from the moisture analysis. After removing a 50-mg aliquot for elemental analyses, the solids and procelain dish are ignited for 30 minutes at 600°C. The resultant ash and procelain dish are cooled in a desiccator and weighed.

The elemental composition method (EPA A003) uses 50 mg of dried solids to determine the percent concentrations of carbon, nitrogen, phosporous, sulfur, and halogens (iodine, chlorine, fluorine, and bromine). Carbon is determined by measuring carbon dioxide and water upon combustion (ASTMD-3178-73). Nitrogen is determined by the Kjeldahl digestion method (ASTM D-3179-73), oxygen by the difference method (ASTMD-2795), sulfur by sulfate titration (ASTMD-3177), and halides by halide titration (ASTMD-2361-66).

Heating value of the sample will be determined using the ASTMD-2015 method. In the method, a one-gram sample is placed in a calibrated isothermal jacket bomb calorimeter under controlled conditions. Calorific values (Btu) will be computed from temperature observations made before, during, and after combustion of the sample.

Viscosity of liquid samples will be determined using the ASTMD-445 method utilizing a kinematic viscometer and a thermometer. The time will be measured for the flow of a fixed volume of liquid through the viscometer.

5.2.23 Volatile Organics in Incineration Off-Gas Samples by Gas Choromatography Mass/Spectrometry (GC/MS)

Due to their volatility, analysis for these compounds will be restricted to incineration off-gas samples collected on Tenax/Charcoal tubes. In this method, the front and back portions of the Tenax/Charcoal tubes are thermally desorbed. These desorbed organics are analyzed by a GC Hall detector using a packed column (1% SP-1000 on Carbopack B). This procedure will analyze for the volatile halo organics. This method will not be certified by PMO, but demonstration will be required to show the detection level that can be achieved.

Other test burns may be used to collect the volatile aromatic organics for analysis, since the tenax traps will permit only one analytical run. The volatile aromatics will be analysed by GC/PID upon the same packed column as described above.

GC/MS will not be used because it does not have the required sensitivity to achieve the Task 17 action levels.

5.2.24 Acid Gases in Incineration Off-Gas Samples

This method was developed by UBTL for the analysis of hydrogen chloride (EPA-600/8-84-001) in incineration off-gases. The method will not be PMO certified.

In the method, 0.2 N sodium hydroxide sorbent from an incineration off-gas impinger is assayed by specific ion probe for the presence of chloride.

5.2.25 Volatile Metals by Inductively Coupled Argon Plasma (ICP) Emission Spectrometry in Incineration Off-Gas Samples

This ICP method, based on EPA method 200.7 (EPA-600/4-79-020), is not PMO certified at the quantitative level. The ICP method has been certified only for soils and waters, not volatile metals.

In this procedure, an aliquot of silver catalyzed ammonium persulfate sorbent is placed in a beaker, treated with concentrated nitric acid and 1:1 hydrochloric acid, and heated to dissolve precipitates that may have formed. The acidified aliquot will be filtered, the beaker rinsed with deionized water, and the rinsate passed through the filter. The digestate is brought to a final volume of 5 ml and assayed by ICP.

5.2.26 Volatile Metals/Arsenic in Incineration Off-Gas Samples by Graphite Furnace Atomic Absorption (AA) Spectrometry

The arsenic method for soils and solids will be developed from EPA Method 7060 (EPA-SW-846). Using this method, an aliquot of silver catalyzed ammonium persulfate sorbent will be digested with hydrogen peroxide and concentrated nitric acid. The digest will be filtered and assayed by graphite furnace atomic absorption spectrometry. The target detection limit for arsenic will be 1 ug/g.

5.2.27 Volatile Metals/Mercury in Incineration Off-Gas Samples by Cold Vapor Atomic Absorption (CVAA) Spectrometry

This mercury method was developed from EPA Method 245.5 (EPA 600/4-82-057). In the method, an aliquot of silver catalyzed ammonium persulfate sorbent will be digested with aqua regia followed by treatment with potassium permanganate. Excess permanganate will be reduced with hydroxylamine sulfate. Mercury will be reduced with stannous chloride and assayed by CVAA. The target detection limit for mercury will be 0.1 ug/g.

5.2.28 Moisture Content in Incineration Off-Gas Samples

The moisture content determinations will not be PMO certified. In this method, the weight of the condensate collected in the trap is measured.

5.2.29 Organophosphorous, Orgonosulfur, and Organochlorine Compounds in Incineration Off-Gas Samples by GC/Selective Detectors

After incineration the bottom residue and fly ash, the XAD-2 sorbent are sohxlet extracted with methylene chloride and the condensate in the liquid trap is extracted with methylene chloride. The extracts are concentrated by Kuderna Danish. The concentrates are then solvent exchanged to isooctane. The final volume will vary from 0.25 ml to 0.50 ml to meet the sensitivity and action levels required of Task 17. This method will not be PMO certified. Demonstration will be required to show the detection level that can be achieved.

The concentrate is analyzed for organochlorine, organophosphorous and organosulfur compounds. The instrumental conditions are the same as those described under the respective sections in Table 5.1-1 for liquid samples.

5.3 ANALYTICAL RESULTS

5.3.1 System Performance Parameters

A run log will be maintained for the bench-scale test unit. This log will note the purpose of a particular test run, the set test conditions, and any abnormalities encountered during the test. The operating parameters such as temperatures, pressures, and flow rates will be recorded on a data sheet. Measurements will be made at appropriate intervals to insure a continuous picture of the operating conditions.

5.3.2 Analytical Results

The concentrations of the constituents measured in the off-gases and solid residues will be analyzed for the original sample volume (for liquids) or weight (for soils). For sludge, the data would be presented based on a dry weight basis.

5.4 CERTIFICATION

The initial feedstock analyses will be performed by PMO certified methods and laboratories for those methods which are currently certified. To reduce the intralaboratory analytical variations and provide the rapid turn around of analyses, HEAI will perform all of the analyses of the individual feed wastes and incineration products except for the physical characterization analyses. No new methods will be certified for this task. However, some methods demonstration will be required. Hittman/Ebasco will use methods approved by PMO but will not perform additional certification analyses. If required to determine the validity of analytical data, qualitative certification would be recommended.

5.5 QA/QC

For Task 17, the sample handling and analytical activities will comply with the established QA requirements stated in the RMA Procedures Manual except

as noted in this test plan. The bench-scale test conditions such as temperature, gas flow rates, pressures, and oxygen levels will be measured using industry-acceptable methods and equipment. These methods will be based on the equipment manufacturers calibration and established procedures.

The analytical procedures for feedstock and solid residues will use QC procedures outlined in the RMA Procedures Manual (Ebasco, 1985). All chemical analyses will include:

- o Calibration standard;
- o Blank; and
- o Matrix spike.

During development of procedures for off-gas and residue chemical analysis, it will be necessary to document the steps used to achieve the required detection limits. The documented procedure will include:

- o Summary of method;
- o Instrumentation and operating conditions;
- o Reagents and materials;
- o Analytes and analytes standard concentration;
- o Details of sample preparation;
- o Calculation; and
- o QC.

For all analyses where the detection or action level is critical, there will be one standard run at two times the required detection limit. The matrix spike also will be at two times the detection level or two times the found analyte concentration.

6.0 EXPECTED RESULTS

6.1 INTRODUCTION

The test program outlined in the preceding sections is designed to develop data at the bench-scale that will ensure success of a full-scale incineration program. It will provide a basis for selecting an appropriate incineration regime and will therefore contribute to the confirmation of the selection of the most desirable technology for waste destruction to a DRE of 99.99 percent. Details of these expected results are outlined below.

6.2 EXPECTED DRE RESULTS

The tests identified above will provide detailed information concerning the ability to remove hazardous organic chemicals from the soils by heating them to temperatures in the 800-1,000°C region. They will determine the extent to which such chemicals as aldrin, endrin, dieldrin, isodrin, and other contaminants can be removed from the soils matrix and put into the vapor state in order to ensure their destruction in an afterburner.

The tests identified above will provide sufficient analytical data to determine the DRE for all organics found in the soils as a function of time, temperature, and oxygen concentration. Specific plots will be as follows:

- DRE as a function of temperature with a residence time of 2 seconds;
- DRE as a function of temperature with a residence time of 5 seconds;
- DRE as a function of time with a temperature of 1,250°C (afterburner);
- DRE as a function of time with a temperature of 900°C (afterburner); and

- o DRE of certain selected POHCs (e.g., aldrin as a function of oxygen concentration for constant time and temperature values.

From these data a rough optimal (most cost effective) regime for destruction of hazardous organics can be developed. This regime will incorporate thermodynamic data concerning the soils (e.g., heat capacity, thermal conductivity, specific heat) plus regime results into conceptual evaluations of fuel consumption (temperature) and equipment volume (residence time) requirements.

DRE values will also be determined for liquid, sludge, and a mixture of wastes at a successful regime associated with soils incineration (e.g., 1,250°C, 2 seconds, 5.4% O_2). Such DRE values will confirm the utility of a selected regime for the entire waste feedstock associated with Basin F.

6.3 EXPECTED TECHNOLOGY SELECTION CONFIRMATION RESULTS

The data above will provide a method for conceptual optimization of the incineration regime to be scaled up from the bench-scale operations to either pilot plant or full-scale operation. These data can then be compared to typical regimes for existing incinerator designs from among the technologies of countercurrent and cocurrent rotating kilns, fluidized beds, and multiple and single hearth furnaces.

In addition to the DRE data described above, the PCT data concerning the soils, sludge, and liquid also will be factored into the evaluation of technologies. Specific issues will include ash fusion temperature of the soils. Such data will be used to determine whether a given technology does or does not have a "fatal flaw" with respect to the wastes found at Basin F. Such data could be used to rule out a given technology if it cannot provide sufficient temperature or residence time to ensure 99.99 percent DRE.

APPENDIX 1

REFERENCES

6.4 OTHER EXPECTED RESULTS

The tests described in preceding sections will contribute by determining initial regimes to be tested either at the pilot-scale or full-scale operation. Further, they will be useful in determining conceptual parameters of a full-scale operation including the following:

Technology	Parameter
Rotary Kiln	Capacity (volume)
	Direction (countercurrent vs. cocurrent)
	Angle and rotational speed for residence time
	Optimal fuel and combustion regime
Fluidized Bed	Capacity (volume)
	Optimal fuel and combustion regime
	Maximum operating temperature
Afterburner	Capacity (volume)
	Optimal fuel and combustion regime

These expected resulted will be essential in developing a cost-effective incineration program for the complete and safe destruction of the hazardous chemicals in Basin F at RMA.

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